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EXPERIMENTAL STUDY OF THE ULTRASONIC EXTRACTION PROCESS OF PLANT RAW MATERIALS

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Abstract: *In-depth scientific research is being conducted around the world aimed at developing the scientific and methodological foundations of energy-saving extractors, processing medicinal plants, increasing the efficiency of modern technologies, processes and equipment for obtaining high-quality pharmaceutical raw materials rich in biologically active substances. Energy-saving extractors, developed in conjunction with the extraction process of medicinal plants, are introduced into the industry using scientifically proven technology. At the global level, special attention is paid to the creation of intelligent designs of innovative extraction plants that operate using ultrasonic waves, allowing the extraction of medicinal components of plants.*
Keywords*: extraction, ultrasound, solvent, cavitation,effective value of sound pressure.*

Annotatsiya: *Jahonda energiya tejamkor ekstraktorlar, dorivor oʻsimliklarni qayta ishlash, biologik faol moddalarga boy boʻlgan yuqori sifatli farmatsevtik xomashyolarni olishning zamonaviy texnologiyalari, jarayonlari va apparatlari samaradorligini oshirishning ilmiy-uslubiy asoslarini ishlab chiqishga qaratilgan chuqur ilmiy izlanishlar olib borilmoqda. Dorivor oʻsimliklarni ekstraktsiyalash jarayoni bilan bogʻliq holda ishlab chiqilgan energiya tejamkor ekstraktorlar ilmiy asoslangan texnologiya asosida sanoatga joriy etilgan. Jahon miqyosida oʻsimliklarning shifobaxsh komponentlarini ajratib olish imkonini beruvchi ultrotovush toʻlqinlaridan foydalangan holda ishlaydigan innovatsion ekstraktsiyalash qurilmalarining oqilona loyihalarini yaratishga alohida eʼtibor qaratilmoqda.*

Ushbu maqolada utratovush ta'siri ekstraksiya jarayonida qo'llanilishi hamda yuqori konsentratsiyali ekstrakt olish usuli ko'rib chiqilgan. Ekstraksiyalash jarayonida erituvchilarni tanlashning ahamiyati tavsiflangan.
Tayanch soʻzlar: *ekstraksiya, ultratovush, erituvchi, kavitatsiya, ovoz bosimining samarali qiymati.*

Аннотация: *В мире проводятся глубокие научные исследования, направленные на разработку научно-методических основ энергосберегающих экстракторов, переработки лекарственных растений, повышение эффективности современных технологий, процессов и оборудования для получения высококачественного фармацевтического сырья, богатого в биологически активных веществах. Энергосберегающие экстракторы, разработанные совместно с процессом экстракции лекарственных растений, внедрены в промышленность по научно обоснованной технологии. На мировом уровне особое внимание уделяется созданию интеллектуальных конструкций инновационных экстракционных установок, работающих с использованием ультразвуковых волн, позволяющих извлекать лекарственные компоненты растений.*

В данной статье рассмотрено использование эффекта элюирования в процессе экстракции и способ получения экстракта высокой концентрации. Описано значение выбора растворителей в процессе экстракции.
Ключевые слова: *экстракция, ультразвук, растворитель, кавитация, эффективное значение звукового давления*

Introduction

The process of separating one or more components from solutions or solids using solvents is called extraction. This process is divided into two types: a) extraction of liquids; b) extraction of solid materials.

The process of extracting one or more components from the composition of solutions with the help of solvents - extractants, which act selectively, is called liquid extraction. When mixing a liquid mixture with a solvent, only the desired components are well soluble in the solvent, while the remaining components are poorly or completely insoluble [1-4].

Extraction processes are also used to separate liquid mixtures, such as rectification. Which of these methods to choose depends on the properties of the substances in the mixture. The rectification process usually takes place under the influence of heat.

Heat is required to perform the extraction. Rectification is based on the evaporation of mixture components at different temperatures. If the boiling points of the components of the mixture are close to each other or they are unstable at high temperatures, the extraction process is used in such cases. The density of the selected solvent must be lower than the density of the liquid to be extracted [3-6].

1. Solvent selection for plant extraction process

Solid-liquid phase extraction is certainly the most widely used method for extracting bioactive components from plant matrices. It consists in removing the solute from the solid matrix using a solvent [2-5].

Table 1 provides an overview of the data for the various materials used.

Table 1

Material information of used solvents

Solvents	Boiling point (ºC)	Density (gsm $^{-3}$)	Vapor pressure (kPa)	Surface tension (mNm^{-1})	Viscosity (mPa)
Water	100	0,9982	31	72,75	1,00
Ethyl alcohol	78,3	0,7893	58	22,55	1,19
Methanol	64,7	0,7914	129	22,60	0,59
Acetone	56,0	0,7902	246	23,30	0,32
Ethyl acetate	77,1	0,9003	97	24.00	0,44

When the solvent interacts with the solid matrix, the following events occur [3,4,6]:

- the solvent spreads from the main part to the surface of the solid matrix;
- the solvent penetrates into the solid matrix through micropores and creates a permanent wetting phase inside;
- the solvent interacts with the solid substance and thereby forms a concentrated solution in the dissolved substance;
- due to the difference in the concentration of dissolved substances between the inner and outer parts of the solid matrix, the concentrated solution spreads to the surface of the solid body;
- diffuses through the boundary layer of the dissolved substance towards the mass of the solution.

Therefore, the process of diffusion and extraction stops when the concentration of solute in the wetting solvent equals that of the solvent outside the solid phase. After reaching such a state of equilibrium, it is possible to proceed with mechanical separation of the solid substance from the solution (pouring, filtering, etc.) [1-3].

This operation may be repeated several times until a satisfactory extraction yield is achieved.

According to the results of our research carried out in the laboratory of the "Pharmaceutical Technologies" department of the "Belarus State Medical University", the mixture of ethyl alcohol and water was considered the most optimal as a solvent for mint and cloves. Because, when analyzed microscopically, many paths of solvent were formed in the matrix of plant pores (Fig. 1).

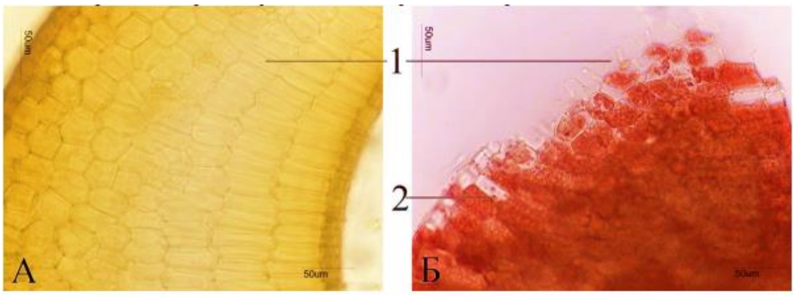

Fig. 1. ***Solvent pathway in plant matrix.***
A) Mint; B) Carnation; 1 - embryonic cells, 2 - fat drops.

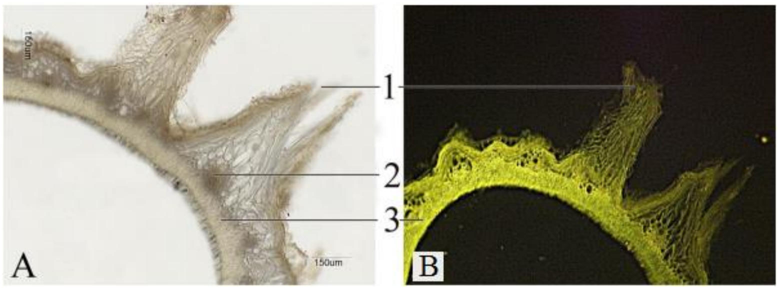

Fig. 2. ***Effect of ultrasound on ethyl alcohol and plant matrix.***
A) Mint; B) Carnation; 1 – melting point; 2 - closed collateral bundle, 3 - sclerenchyma belt.

In addition, the properties of the solvent can be increased or accelerated by ultrasound. When we analyzed the solvents listed in Table 1 in the laboratory of the "Pharmaceutical Technologies" Department of the "Belarus State Medical University", it was the ethyl alcohol (Fig. 2) that was selected as the most optimal process.

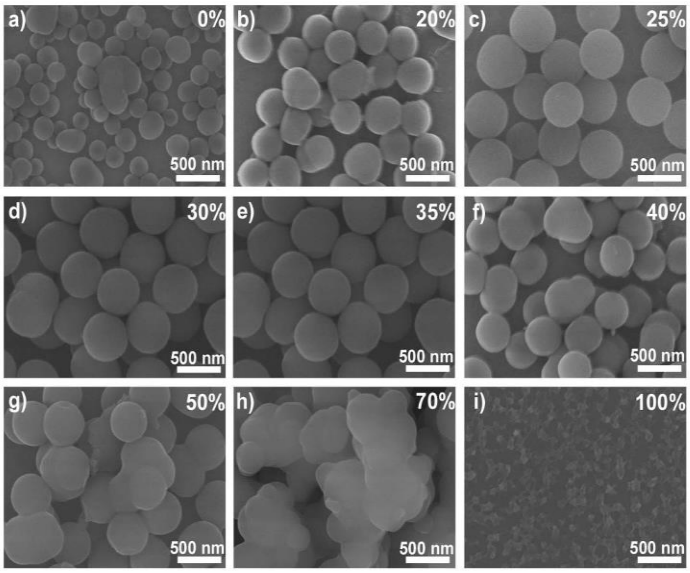

Fig. 3. ***Water-alcohol mixture.***
a) 0%; b)20%; c) 25%; d) 30%; e) 35%; f) 40%; g) 50%; h) 70%; i) 100%.

Water and alcohol are versatile solvents, and the water-alcohol system is widely used. Fig. 3 shows the results of experiments conducted in water-alcohol mixed solvents. Experiments were conducted in various water-alcohol mixed solvents to investigate the effect of the solvent.

In short, plant raw bioactive substances dissolve well in a solvent containing 70% alcohol in a water-alcohol mixture. Alcohol solvent above 70% is expensive and has a slow effect on the dissolution of macro and micronutrients in plant raw materials.

2. Study of the effect of ultrasound on the extraction process.

The effectiveness of using ultrasound can be explained by the influence of a number of specific factors characteristic of ultrasound vibrations [1].

In extraction, ultrasound can be used to reversibly or irreversibly create pores in the cell membrane, allowing cell contents to be released into the extractive medium. We investigated the use of ultrasound at a frequency of 30-50 kHz for the extraction of peppermint. Figure 3.6 shows a comparison between untreated mint (Fig. 4-A) and ultrasonically extracted mint (Fig. 4-B) [1,2].

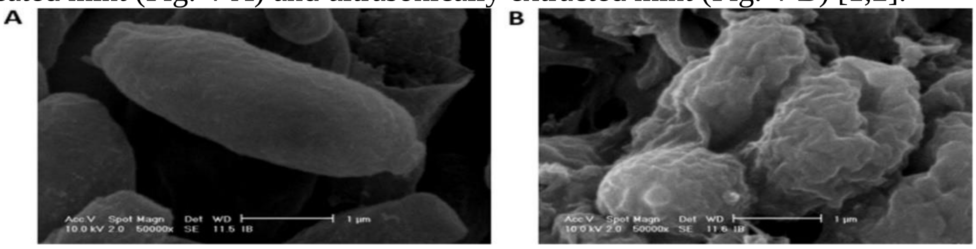

*Fig 4. **The effect of ultrasound on the mint plant.***
A - Microscopy of mint; B - Microscopy of mint extracted under the influence of ultrasound.

During ultrasonic extraction, shear forces occur in the liquid and in close proximity to the plant material. Shear forces and turbulence are caused by the vibration and disruption of a cavitation bubble in a liquid [1-4]. Extraction kinetics of peppermint plant are compared between ultrasound and cavitation (Fig. 5-1).

Extraction of peppermint plant shows direct dissolution, while with ultrasound the extraction process appears more diffuse (Fig. 5-3).

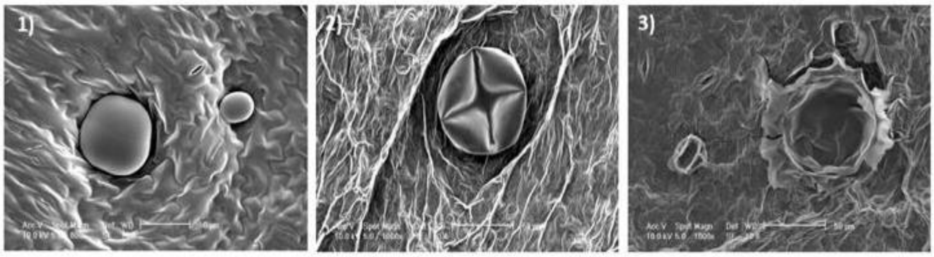

*Fig. 5. **Microscopic observation of the effect of ultrasound power on mint leaves.***
1) before extraction, 2) when exposed to 30kHz ultrasound and 3) when exposed to 50kHz ultrasound.

Research shows that the use of ultrasound not only significantly accelerates the production process, but is also effective compared to other methods of obtaining the main product. The increase in extraction efficiency is related to the increase in the mass transfer coefficient and the volume of the intermediate surface.

When the extraction process is exposed to ultrasound in a liquid medium, a variable sound pressure is created that helps the extracted substance penetrate into the cracks and capillaries. The intensity of the extraction process and the diffusion coefficient depend on the values of the amplitude and frequency of the forced vibrations of the liquid.

At the edge of an open microcrack, microflow turbulation occurs during intense movement of the liquid. Here, the solid phase dissolution process is limited by the turbulent diffusion coefficient. Turbulent flows from the first zone to the second zone carry out the transfer of the main part of the obtained substance. Mass transfer in the third zone depends on chaotic molecular motion. Longitudinal and transverse dimensions of microcracks are an important factor in the melting process.

Ultrasound, which creates a capillary effect, not only accelerates the movement of bubbles, but also creates conditions for its dissolution in liquids. As a result of the creation of a vacuum, the time of soaking raw materials under the influence of ultrasound is significantly reduced. At normal temperatures (0-25°C), the solubility limit with ultrasound is increased in the range of difficult and practically insoluble substances, and the saturation concentration can exceed the known constants by 5-30 times [1, 2].

UZTA-0.4/22-OM "Volna" ultrasound technological apparatus was used for measuring ultrasounds for the research (technical characteristics are listed in Table 2). The principle of operation is based on the use of the characteristics of high-intensity ultrasound vibrations in liquid and liquid-dispersed media, and the ultrasound mode for the extraction process was determined: frequency (30-50±1.65) kHz, power 180 W, exposure 5 min. The ratio of solvent and extract was set the same for all studied samples.

Table 2

Technical parameters of the УЗТА-0,4/22-ОМ device

Name of the indicator	Value
Mechanical vibration frequency, kHz	30-50±1,65
Power, W	400
Power control range, %	30–100
Excess pressure of the processed medium, atm	4

The choice of the method of ultrasound exposure is based on studies to determine the nominal value of sound dB. We used the MEGEON 92135 device (Fig. 6) to determine the dB level of ultrasound.

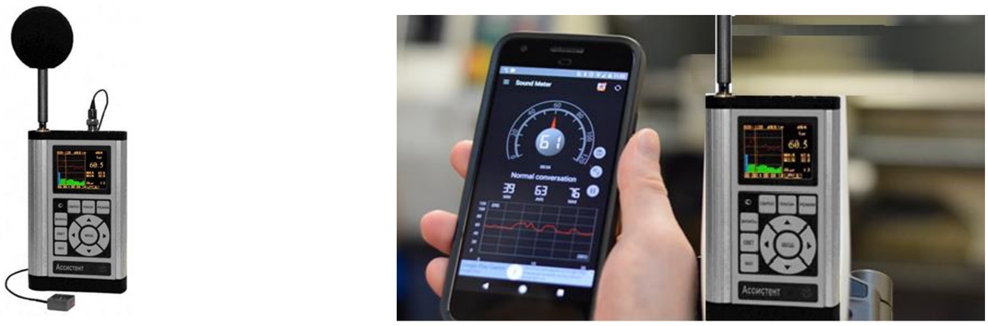

Fig. 6. ***MEGEON 92135.***

The frequency can be selected by determining the nominal value of the sound in dB. Because we can know the range of the influence through noise. In the experiment, the noise was measured in 4 ways, i.e., with a frequency of 30 Hz in the presence of solvent in the Soxhlet (Fig. 8) and in the absence of solvent (Fig. 7) and in the presence of solvent in the Soxhlet with a frequency of 50 Hz (Fig. 9) and in the absence of solvent (Fig. 10) was studied by influencing.

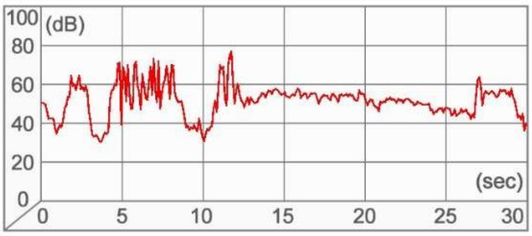

Fig. 7. ***The result obtained under the influence of 30 Hz vibration in the absence of a solvent in a Soxhlet.***

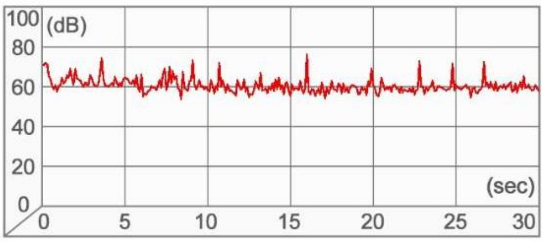

Fig. 8. ***The result obtained under the influence of 30 Hz vibration in the presence of a solvent in a Soxhlet.***

Analyzing Figures 7 and 8, it was found that the most nominal dB when exposed to 30 Hz is 45-50 dB without the solvent in the socket, and 60 dB when the solvent is present. It can be seen that in the presence of a solvent, the range of influence of ultrasound is higher.

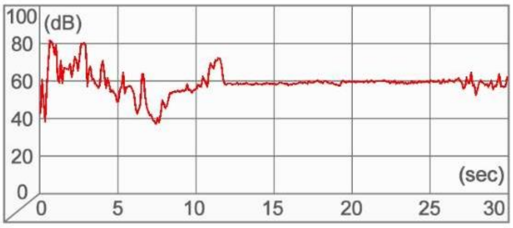

Fig. 9. ***The result obtained under the influence of 50 Hz vibration in the absence of solvent in the soxhlet.***

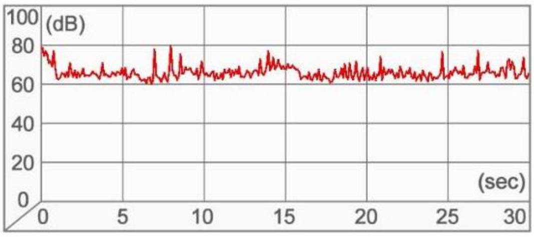

Fig. 10. ***The result obtained by exposure to 50 Hz in the existing state of the solvent soxhlet.***

When exposed to 50 Hz (Figures 9 and 10), the most nominal dB was found to be 60 without the fuse in the socket, and 70 dB with the fuse. It can also be concluded that the effect of ultrasound is high in the presence of a solvent.

Ultrasonic vibration systems work effectively only in the resonant mode, that is, at the resonant frequency of the generator. In almost all automatic frequency control (AFC) systems of electronic generators, the criterion for adjusting the resonance frequency is the zero phase shift between the current and voltage in the piezoceramic elements of the emitter.

Figure 11 shows the amplitude-frequency characteristic of the ultrasonic vibration system (curve 3), the frequency dependence of the mechanical network current (the current consumed by the emitter, the static capacitance of the piezoceramic (curve 2)) shown. As a mechanical vibration system, the phase-frequency characteristic was obtained as a result of measuring the changes between the current and voltage in the piezoceramic elements obtained at different values of the real phase-frequency characteristic (curve 1) and statics of the vibration system.

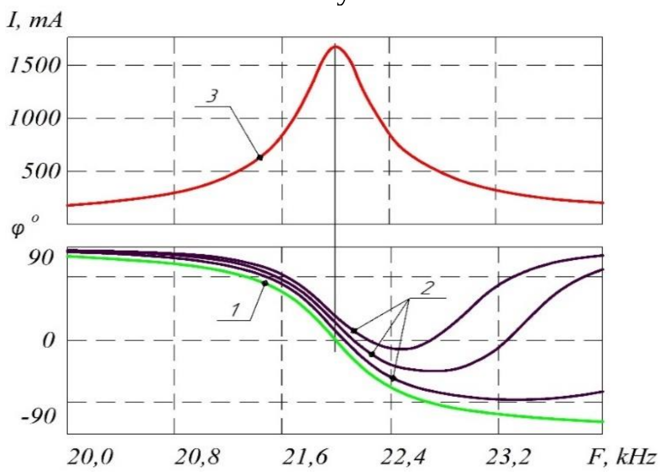

Fig. 11. ***Amplitude-frequency and phase-frequency characteristics of the piezoceramic element depending on the change in electric capacity.***

These curves were obtained using an equivalent electrical model of an ultrasonic emitter with a resonance frequency of 30-50 kHz. These relationships are presented as an example to show the influence of the electric capacitance of piezoceramic elements on the frequency dependence of the phase shift between current and voltage in the emitter (curve 2).

Conclusion

From the presented relationships, it follows that the change in the electric capacity of the piezoceramic elements due to the heating during the operation of the emitter, the AFC system of the existing ultrasonic generators leads to the orientation of the phase shift between the current and the voltage in the piezoceramics. As mentioned above, the operation of the ultrasonic vibration system in a liquid medium is accompanied by a strong heating of its structural elements, which not only leads to a decrease in the resonance frequency, but also to an increase in the electric power of the piezoelectric elements by 1.5. With a high quality factor of the system, due to errors in the operation of the AFC system (caused by the effect of the capacitance of the piezoceramic elements), the intensity of the ultrasound effect and the efficiency of the performed processes decrease.

In order to eliminate the influence of the piezoelectric emitter and the sound generator on the frequency matching with the ultrasonic vibration system, a scheme for accepting the periodic operation of the generator was proposed and developed, thus its phase-frequency characteristic is repeated. As a mechanical vibration system, the phase-frequency characteristic of the emitter does not depend on the electric power of the piezoceramic elements.

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