

Green Approach to Intensify the Extraction Processes of Substances from Plant Materials

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ABSTRACT

The principles of sustainable development and green chemistry are priorities in the development of new technologies for the extraction of useful biologically active substances, for example, in the manufacture of medicines. The processes of extracting substances from plant raw materials have many applications. Intensification of such processes, reducing their duration, energy consumption and consumption of the extractant is urgent. The object of the research is the green approach to intensify the extraction processes of polysaccharide substances from plant materials. The research methodology was experimental with statistical data processing. In the field of experimental research was the process of the influence of pulsed, alternating and direct current on the intensity of aqueous extraction of polysaccharides from plant materials. The high efficiency of intensification of extraction by a pulsed electric field was studied. The process can reduce the time for obtaining water-soluble polysaccharides by 3 times in comparison with traditional pharmacopoeial convection methods and reduce energy consumption by 20 times. The significance of the pulse current parameters for effective extraction was determined: the referred pulse shape, the frequency of the pulse current for extracting the maximum amount of water-soluble polysaccharides, the pulse duration, and the optimal processing time. The green approach to the use of a moderate electric field of pulsed current to accelerate the extraction of biologically active substances is the basis of the method of intensification of other traditional mass transfer processes for the sustainable development of technology.

Keywords: green approach, plant materials, extraction, polysaccharides, pulsed electric field.

INTRODUCTION

The search for the methods to intensify the release of natural substances of different structural organization for comprehensive practical application on the principles of green chemistry, along with finding new and improving the already known methods of synthesis is an urgent task of both applied and fundamental sciences. At the base of modern green technologies, which are

aimed at achieving zero waste, reducing energy costs, are the principles of sustainable development (Ishchenko and et al., 2019; Mariychuk et al., 2020a; Mitryasova et al., 2017, 2020; Bezsonov et al. 2017; Pohrebennyk et al., 2016). Evaluation of the environmental impact of a chemical process requires the consideration of numerous factors, such as processing efficiency, safety, and cost effectiveness (Eissen, 2012; Hutchison, 2019; Mitryasova et al., 2017; Petrov

et al., 2020). These criteria have historically been the primary consideration of the development of new chemical processes. The green chemistry principles also demand a detailed analysis of environmental and health impacts (Mariychuk et al., 2020b; Yushchishina et al., 2020).

The principles of sustainable development and green chemistry are a priority in the development of new technologies for the extraction of useful biologically active substances, for example, in the manufacturing of medicines. Biologically active substances have a wide variety of unique physiological functions and are used in many areas of medicine, pharmacy, and the food industry. The isolation of these substances from plants is the focus of many studies, namely the factors that contribute to the intensification of extraction processes. *Hyssopus officinalis* (*Hyssopus officinalis* L.) is a perennial shrub of the goose family, has long been used in folk medicine as an expectorant and anti-inflammatory agent for the diseases of the upper respiratory tract. Hyssop herb infusion is used to treat hard-to-heal wounds. the review (Sensoy & Sastry, 2004; Kaur et al., 2016; Tahir and al. 2018; Granot & Rubinsky, 2008; Kulshrestha et al., 2008), which summarizes all the published scientific works on this medicinal plant over the past two decades, stands out among the scientific publications on the phytochemical and pharmacological profile of a medicinal plant. It has been shown that *Hyssopus officinalis* L. is a very important culinary and medicinal perennial plant, widely cultivated in Asia, Europe and America.

Hyssopus officinalis contains numerous phytochemicals, exhibits antioxidant, anticonvulsant, antifungal, antimicrobial activity, as well as has antihemolytic, antiulcer, and spasmolytic effects. Hyssop essential oil is widely used in the cosmetic, food and pharmaceutical industries all over the world, it is especially often used in sore throat phytotherapy. Thus, *Hyssopus officinalis* L. is an important medicinal plant that can be used to treat several diseases such as microbial infection, epilepsy, ulcers and spasms of various etiologies. The methods for extracting biologically active substances from plant materials do not always allow extracting target substances with a high yield, in accordance with the principles of green chemistry and sustainable development. Therefore, the question of the intensification of the processes of extraction of biologically active substances remains relevant.

The separation of mixtures in the extraction process with a specific solvent usually takes place at room temperature. It is possible to increase the yield percentage of the target component under the influence of various factors on the extraction process. For example, the effect of various kinds of electric fields is little studied. Thus, the hypothesis of the study is the possibility of increasing the speed of the extraction process and the completeness of the extraction of target components using electric current. Achieving this goal will improve the quality of plant raw materials, rationally use natural resources, reduce energy costs and implement the principles of green chemistry.

The object of the research is the green approach to intensify the extraction processes of polysaccharide substances from plant materials. Extraction methods, depending on the properties of the target components, are divided into methods of isolation and distillation. In addition, extraction methods are divided into static and dynamic. The latter consist in the constant change of the extractant and raw materials relative to each other, they are more efficient than static ones, since the extraction of substances in them occurs due to convective diffusion. (State Pharmacopoeia of Ukraine, 2007). In the production of tinctures, classical methods of maceration or infusion (Bukeeva and Kudaibergenova, 2012), as well as countercurrent method (Wang & Curtis, 2006), percolation method (Luque de Castro & Garcia-Ayuso, 1998), and Soxhlet method are used. (Richter et al. 1996; Suna et al. 2012). Liquid extraction, presented in the works (Kulazinsky et al., 2016), is a more modern method for extracting biologically active substances from plant materials. In addition, the methods of accelerated supercritical fluid extraction (Awaluddin et al., 2016), subcritical water extraction, and extraction using ultrasound are quite fully described.

The ultrasonic method, along with a significant increase in the extraction rate of the target products, also makes it possible to increase their yield. The ultrasonic method enables to achieve good results in the extraction of target components due to cavitation processes (Chemat et al., 2004). Cavitation allows intensifying diffusion, which changes the pH of the medium and the splitting of macromolecular compounds. The ultrasonic method showed very good results in the process of extraction of bioorganic compounds from plant materials, such as fats, esters (Cravotto et al., 2004) and food additives from plants.

At the same time, the ultrasonic method also has disadvantages, which consist in the destruction of the target components. Modern research has determined the exact parameters of ultrasonic extraction, which can lead to the destruction of bioorganic molecules. Among the modern physical methods for intensifying the processes of extracting target components from plants, the following can be identified: microwave; magnetic pulse; electric discharge (Kanduser and Miklavcic, 2008; Mandal et al., 2007; Varghese et al., 2014). Some biological methods have also found application (Malushevsky and Yushchishina, 2001; Malyushevsky et al., 2017).

EXPERIMENTAL METHOD

The conducted research on the method of intensifying the extraction of polysaccharides from plant raw materials was aimed at processing the dry plant raw material / solvent suspension, as the processing of freshly harvested plant objects requires complex logistics and is difficult to scale to modern processing plants. However, from the point of view of understanding the mechanism of interaction of the electric current that generates the electromagnetic field and affects the plant raw material / solvent system, an experiment with freshly harvested hyssop (*Hyssopus officinalis* L.) was also necessary. Therefore, several samples of the suspension were also prepared from freshly harvested hyssop in terms of weight of dried grass (Trineeva and Slivkin, 2017).

Drying was carried out immediately after harvesting. In freshly harvested plants, biochemical processes continue, which take place under the influence of enzymes, the activity of which not only stops when plants wither, but passes vigorously due to the humid environment and elevated temperature. Tap water used for the preparation of the suspension had the following characteristics: hydrogen index – 6.7, the total number of insoluble particles – 320 ppm, specific conductivity – 0.45 $\mu\text{Sm}/\text{cm}$, redox potential – 268 mV. In the course of the experiment, the changes of these parameters for the obtained extracts were investigated (Malyushevskaya et al., 2021). Control samples of polysaccharides aqueous extract from hyssop («Control») were prepared according to (State Pharmacopoeia of Ukraine, 2007). The modes of pulse current / raw material / suspension suspension were varied in terms of

pulse frequency, and the voltage was varied for alternating and direct current suspensions, in all cases the same amount of energy was applied to the volume of suspension ($E \approx 1 \cdot 10^4 \text{ J}$).

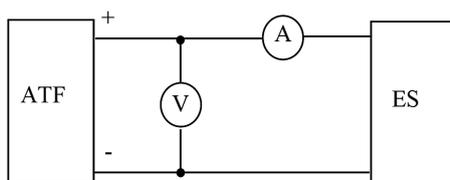
The picric method was used as a method to assess the efficiency of the extraction of water-soluble polysaccharides from plant raw materials under the influence of electromagnetic fields, which are formed as a result of different types of current flow (Trineeva & Slivkin 2017). This method made it possible to relatively quickly determine the quantitative content of the sum of polysaccharides in the samples of the obtained extracts. The method is based on the color reaction of monosaccharides with picric acid with the formation of aminocaproic acid as a result of the reduction of the NO_2 group in NH_2 by sugar. The resulting reduced monosaccharides with picric acid in an alkaline medium have an absorption maximum in the range of 440–460 nm. In the considered case, the essence of the measurement was to compare the light transmission of the reference liquid sample and the sample under study at a wavelength of 455 nm. In parallel, the optical density of a standard glucose sample, processed similar to the test sample of the solution, was measured.

The content of the sum of free sugars C in the test sample in terms of glucose in dry raw materials in percent was determined by formula 1:

$$C = \frac{5 \cdot C_{gl} \cdot D}{D_{gl}} \quad (1)$$

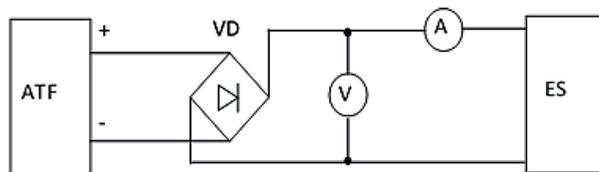
where: D is the optical density of the test solution, relative units; D_{gl} is the optical density of a solution of a standard glucose sample, processed similarly to the test solution, relative units; C_{gl} is the glucose content in a standard sample, g/l; coefficient 5 takes into account the races -creation of the derivative extract 5 times.

The optical densities of the obtained filtrates were determined using an SF-56 spectrophotometer (TC 3-3.2367-91). To generate rectangular pulses, two parallel-connected sources (single-phase and three-phase) were used as a pulse current generator AC processing was carried out using the equipment, the diagram of which is shown in Figure 1. DC processing was carried out using equipment, the diagram of which is shown in Figure 2. Figure 3 shows a schematic electrical diagram of a low-power pulse current generator. Through the



ATF – autotransformer; ES – electrode system; V – voltmeter; A – ammeter

Fig. 1. Diagram of experimental equipment for AC processing



ATF – autotransformer; ES – electrode system, VD – diode bridge; V – voltmeter; A – ammeter

Fig. 2. Diagram of experimental equipment for DC processing

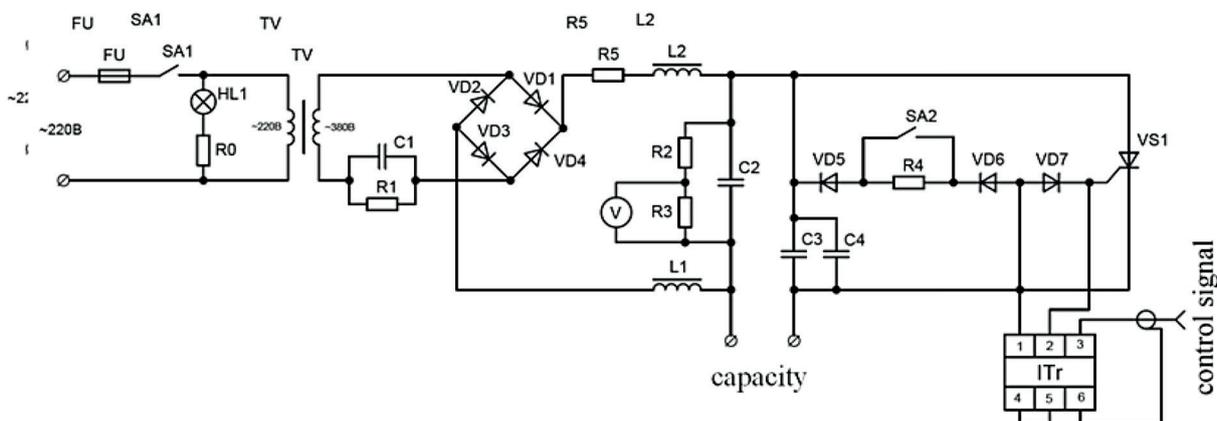


Fig. 3. Schematic electrical diagram of a single-phase current pulse generator

step-up transformer TV and rectifier VD1-4 capacitor C2 is charged to a voltage of 480 V (at a pulse frequency of up to 40 Hz). To eliminate the disadvantages of a single-phase low-voltage pulse source, namely the decrease in the amplitude of the current pulse due to the increase in the frequency of the pulses, it was proposed to charge capacitors from a three-phase power supply circuit.

The generator can operate in the mode of forming a bipolar pulse, which is a damped sine wave with an amplitude of I_{max} , the period of oscillation 30 μ s. Technical data of the three-phase low-voltage current pulse generator:

Power supply parameters:

- mains voltage 380 V;
- network frequency 50 Hz;
- maximum power consumption 13.5 kVA;
- rated power 12 kW;
- operating voltage 515 V.

RESULTS AND DISCUSSION

The plant/tap water suspension was treated with electromagnetic field currents of various types. Analysis of the results obtained from the extraction of water-soluble polysaccharides from hyssop herb by various methods is presented in Figure 4. The extract by the original extract (“Zero”, $C = 0.3643 \text{ g/dm}^3$), which was obtained by pre-soaking dry hysop herb in water and further infusion at room temperature within 12 hours is occupied the lowest position in the content of polysaccharides.

The extract obtained from the herb hyssop medicinal following the settings of the State Pharmacopoeia using prolonged (25 min) heating of a suspension of dry herb hyssop/water in a water bath and further infusion at room temperature (sample “Control”) contains 1.3196 g/dm^3 of water-soluble polysaccharides – that is, the

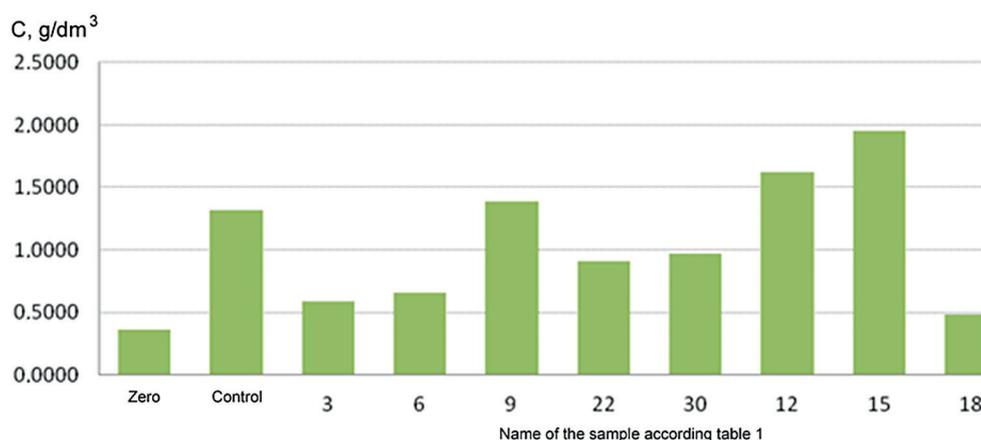


Fig. 4. Polysaccharides content in the investigated extracts

content of soluble polysaccharides increased 3.62 times. The extract “15” is characterized by a high content of polysaccharides, obtained after a short (475 s) pulse treatment with an electric current of a suspension of hyssop herb/water at a frequency of 500 Hz and further infusion.

When comparing the content of polysaccharides in this extract with the extract of the original sample “Zero”, an increase in the content of target substances in the extract by 5.4 times can be seen, while at the end of the processing, the temperature of the suspension was only 40°C exclusively due to Joule heating. The concentration of polysaccharides in the extracts obtained by processing with various types of current is a criterion for the effectiveness of processing. Processing with a pulsed current is the most effective.

Treatment of a suspension of hyssop grass – water with currents of other types (sample “22” - alternating current, $C = 0.9098 \text{ g/dm}^3$; sample “30” – direct current, $C = 0.9665 \text{ g/dm}^3$), led to an increase in the concentration of polysaccharides in the extracts, only three times as compared to the initial one, even though the energy introduced into the suspension utilizing alternating and direct currents remained approximately equal to the energy of pulsed current processing. The maximum content of soluble polysaccharides (1.9524 g/dm^3) shows the extract “11+”, obtained after treatment of the suspension with pulsed current at a generator frequency of 500 Hz. Oscillograms of the processing process are shown in Figure 5. The obtained result attracted the authors’ attention, so to determine the effect of pulsed current on the extraction processes, the experiment was repeated while maintaining unchanged all other parameters of the pulse, except for its duration.

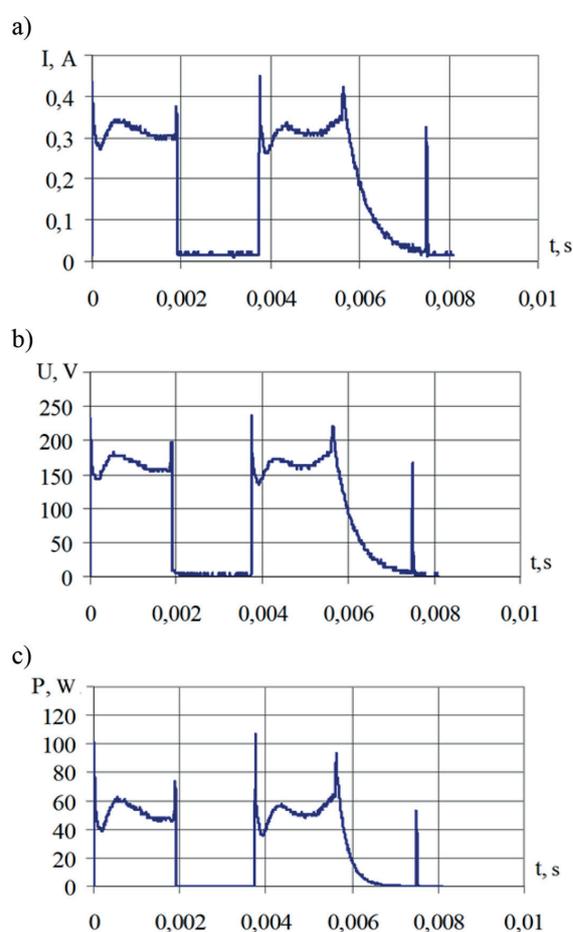


Fig. 5. Oscillograms of current (a), voltage (b) and power (c) when processing the sample “11+” suspension plant/water pulses at a generator frequency of 500 Hz at a temperature of 40°C

It turned out that the pulse duration is of great importance for the intensification of extraction processes. When it was reduced by half, the treatment of the suspension with current was much less effective (in the obtained extract (sample “1+”),

despite the fact that the total energy introduced into the treated volume ($1.25 \cdot 10^4 \text{ J}$) is close to the energy used to obtain sample “11+” ($1.37 \cdot 10^4 \text{ J}$), the content of water-soluble polysaccharides is only 0.5909 g/dm^3 .

To determine the energy required to obtain extracts of plant polysaccharides by means of various methods, an experiment was conducted to study the effect of temperature (from 40°C to 90°C , in steps of 10°C) on the yield of target products. The best result is sample “15” (the suspension temperature is 70°C , electricity consumption – $28.2 \cdot 10^4 \text{ J}$, the pulsed electric current treatment – $1.37 \cdot 10^4 \text{ J}$). From the viewpoint of green approach, the following factors were studied in this research: the yield of the target product, energy consumption, the influence of pulsed electric current. The method of pulsed electric current has shown a significant advantage over heating, and has a twenty-fold advantage. In the future, it is important to study the effect of this method on extractants of chemical nature.

The fundamental possibility of using electric current has been studied. The results of the work testified to the principle advantage of pulsed electric current for intensification of extraction processes of target components from plant raw materials. A series of experiments with samples of aqueous plant extracts made it possible to determine the optimal parameters for the highest yield of the product (polysaccharides). Such parameters are pulsed electric current with voltage $E \leq 100 \text{ V/cm}$ and a fairly short processing time $t = 400\text{--}500 \text{ s}$. Thus, the practical application of the principles of green chemistry (reduction of energy consumption, zero level of waste) during the extraction of polysaccharides from plant raw materials is the use of pulsed electric current. It has a number of advantages, because it occurs at lower temperatures and gives a higher product yield.

CONCLUSIONS

The focus was on the study of constant, variable and pulsed types of electric fields for extraction processes. In the research, the influence of pulsed electric current on the extraction processes of the target product (polysaccharides) from medicinal plant raw materials was studied. The fundamental possibility of applying this method was proven. The influence of different parameters of pulsed electric current on the content of target

components (polysaccharides) in the aqueous extract of plant raw materials has been studied.

In comparison with the traditional pharmacopoeial method, the conditions of pulsed electric current treatment have demonstrated the possibility of increasing the content of extracted polysaccharides by 48% after extraction. The process enables to reduce the time for obtaining water-soluble polysaccharides by 3 times in comparison with traditional pharmacopoeial convection methods and reduce energy consumption by 20 times. The significance of the pulse current parameters for effective extraction was determined: the referred pulse shape, the frequency of the pulse current for extracting the maximum amount of water-soluble polysaccharides, the pulse duration, and the optimal processing time.

The pulsed electric current and conditions for carrying out the processes of extraction of target substances, such as the temperature of the extraction processes (low final temperature up to 40°C), make it possible to achieve the maximum yield of substances while maintaining their biological activity during extraction. At the same time, they enable achieving maximum energy savings, which allows stating that the green approach was implemented.

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