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FACULTY OF BIOTECHNICAL SYSTEMS ENGINEERING
BIOTECHNICAL SYSTEMS ENGINEERING DOCTORAL SCHOOL

DOCTORAL THESIS - ABSTRACT

RESEARCH ON IMPROVING THE PERFORMANCE OF EXTRACTION EQUIPMENT FOR ACTIVE PRINCIPLES FROM MEDICINAL AND AROMATIC PLANTS

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Symbols and notations

Q	volumetric flow rate of the fluid [m^3/s]
K	hydraulic conductivity of the porous material [m/s]
A	cross-sectional area through which the fluid flows [m^2]
$\frac{dh}{dl}$	hydraulic gradient, representing the change in head along the length of interest
dQ	volume of percolated liquid over a time interval, [m^3]
P_p	permeability coefficient of the porous medium [m/s]
I	hydraulic gradient
h	height of the liquid column determining percolation [m]
l	length of the permeable material column [m]
A_m	cross-sectional area of the material sample [m^2]
p	pressure [Pa]
F	applied force [N]
A_c	contact area [m^2]
k	permeability of the porous medium [m^2]
L	length of the porous medium in the flow direction [m]
Δp	pressure difference [$\frac{kg}{(m \cdot s^2)}$]
η	fluid viscosity [$\frac{kg}{m \cdot s}$]
J	diffusion flux [mol/m^2·s]
dm	variation in the quantity of substance [mol]
D	diffusion coefficient [m^2/s]
C	particle concentration [mol/m^3]
x	position coordinate [m]
R	bubble radius [m]
$\dot{R}$	rate of change of bubble radius [m/s]
$\ddot{R}$	acceleration of bubble radius variation [m/s^2]
p_g	partial pressure of the non-condensable gas [Pa]
p_v	partial pressure of the vapor [Pa]
μ	liquid viscosity [Pa·s]
p_0	static ambient pressure [Pa]
$p_s(t)$	instantaneous acoustic pressure [Pa]
ρ_0	equilibrium density of the liquid [kg/m^3]
σ	surface tension at the liquid–gas interface [N/m]
c_∞	sound speed in the liquid under ambient conditions [m/s]

p_∞ ambient pressure and liquid density considered constant under ambient conditions ($\rho=\rho_{L,\infty}$ = const.) [Pa]
p_B pressure of the liquid at the bubble wall [Pa]
R_{max} maximum bubble radius [μm]
R_0 initial bubble radius [μm]
$\upsilon_{microjet}$ microjet velocity [m/s]
υ_{colaps} bubble collapse velocity [m/s]
τ_w wall shear stress [Pa]
U liquid velocity at the wall [m/s]
y distance to the boundary [m]
μ_a dynamic viscosity of water [10^{-3} Pa·s]
UI ultrasonic intensity [W/cm^3]
P ultrasonic power [W]
V sample volume [cm^3]
Y predicted response value
X independent factor
b_1, b_2, b_3 regression coefficients for the linear effect
b_{11}, b_{22}, b_{33} coefficients for the quadratic effect
b_{12}, b_{13}, b_{23} coefficients for the linear interaction effect
μ_m overall mean of observations
α_i effect associated with level i of factorα
β_j effect associated with level j of factorβ
$(\alpha\beta)_{ij}$ interaction effect between levels i and j of factors α and β
ε_{ijk} random error of the model
X_i , X_j values associated with analyzed factors
β_0 constant term
β_i , β_{ii}, β_{jj} coefficients expressing the main effect of each variable
β_{ij} coefficients reflecting interaction effects between variables
ε residual error
n number of studied factors
Ni level of each factor
α_1, α_2, α_3 coefficients of the individual effects
α_{12}, α_{13}, α_{23} coefficients of binary interaction effects
α_{123} coefficient of ternary interaction effect
D_{eff} effective diffusion coefficient [m^2/s]
C_{sol} solute concentration in solid [mg/g dm]
x_d axial distance [m]
t time [s]

$C_{e\,sol}$	equilibrium content of active compounds in the solid phase
Bi	relative importance of internal and external resistances to mass transfer [h_m, m/s]
L_p	half thickness of the plate [m]
D_0	pre-exponential factor [m^2/s]
E_a	activation energy [J/mol K]
R_g	gas constant [8,31 J/mol K]
T	extraction temperature [°C]
Pot	ultrasonic power density [W/L]
ε_p	porosity
S_0	specific storage coefficient
Dk	molecular diffusion
T_0	initial temperature
Λ	hydrodynamic–thermal conductivity tensor
$C_m{}^s$	unknown concentration
$\rho^s c^s$	volumetric heat capacity of the solid
ρc	volumetric heat capacity of the fluid
$R_m{}^s$	total reaction rate
ε_s	solid volume fraction
χ, f_μ	coefficient of floatability and viscosity function
K_h	hydraulic conductivity tensor
e	unit coordinate vector
q	Darcy flux
R_k	total reaction rate of species k
j_k	hydrodynamic dispersion
Y_c	extraction concentration of the compound
B	solvent-to-solid ratio [mL/g]
X_{SA}	amplitude of sonication [%]
X_{PC}	pulse cycle [s]
C_p	total content of active compounds (polyphenols) [mg GAE/100g]

CHAPTER 1
INTRODUCTION

1.1. Presentation of the research topic and justification of its approach

The doctoral thesis titled "*Research on improving the performance of extraction equipment for active principles from medicinal and aromatic plants*" aims to contribute to the development of the field by improving the equipment used in the extraction process, based on the analysis of operational parameter variability. The research integrates advanced extraction methods, especially hybrid technologies, analyzing the performance of the equipment in various configurations and identifying parameter combinations that ensure stable, and efficient operation.

1.2. Importance and relevance of the research topic

The motivation for selecting this topic lies in the need to improve industrial equipment used to obtain active principles under conditions of high yield, reduced costs, and minimal environmental impact. This aspect is particularly relevant as natural resources become increasingly limited, and mechanical engineering must develop advanced technologies to meet these challenges. The novelty of this work is based on the use of a hybrid extraction method: ultrasound-assisted pressure percolation, which combines the benefits of percolation, providing a constant and uniform extraction, with those of ultrasound, which amplifies the process through the action of ultrasonic waves.

1.3. Presentation of the general and specific objectives of the thesis

The general objective of the thesis "*Research on improving the performance of extraction equipment for active principles from medicinal and aromatic plants"* is to increase the performance of extraction equipment used in processing medicinal and aromatic plants by improving technological parameters and integrating percolation under pressure assisted by ultrasound to achieve high yield and energy efficiency. The scientific activity of this doctoral thesis was carried out gradually through the following specific objectives:

- Ensuring the instrumentation and measurement devices required for experimental studies on the extraction of active principles.
- Establishing experimental variants for selecting and configuring the extraction equipment.
- Determining the characteristics of the medicinal plant materials used in the experiments.

- Selection of the plant raw materials used in the experiments: nettle, lavender, and sage, species with different chemical and structural profiles.
- Identification of the optimal cutting degree and the required quantity of plant material for the experiments.
- Study of the influence of technological parameters on extraction efficiency, with controlled adjustment of these parameters.
- Processing and interpretation of experimental data to highlight the relationships between process parameters and extraction yield.
- Mathematical modeling of the extraction process.

1.4. Structure of the doctoral thesis and concise presentation of the chapters

The thesis is structured into seven chapters, developed over 147 pages, containing 108 figures, 38 tables, and 49 mathematical relations, along with a bibliography consisting of 185 references.

- **Chapter 1** *"Introduction"* presents the research topic, its justification, and its importance. The work aims to improve the performance of extraction equipment for active principles from medicinal and aromatic plants by improving process parameters and using hybrid methods, such as percolation under pressure assisted by ultrasound. The chapter defines the hypothesis, the general and specific objectives, the technical challenges, and the experimental methodology, which combines theoretical and practical analysis to achieve efficient, stable, and sustainable extraction processes.
- **Chapter 2** "*Considerations on medicinal and aromatic plants. Environmental factors. Active principles*" presents the general characteristics of medicinal and aromatic plants (nettle, lavender, and sage). It also analyzes the environmental factors that influence the development of these species, the main bioactive compounds, and the essential categories of active principles present in their structure.
- **Chapter 3** "*Extraction technologies and equipment for obtaining active principles from medicinal and aromatic plants*" presents modern extraction methods for active principles (including percolation under pressure assisted by ultrasound) as well as classical methods used in practice, offering a description and comparative analysis of these technologies.
- **Chapter 4** "*Current state of research on the extraction process of active principles from medicinal and aromatic plants*" is structured in two parts.The first part presents recent studies focused on theoretical research related to statistical modeling applied to the optimization of extraction methods, using techniques such as response surface

methodology, full factorial design, and fractional factorial design.The second part provides an overview of current experimental research, highlighting the application of these statistical methods and the obtained results.

- **Chapter 5** "*Experimental research on improving the performance of equipment for the extraction of active principles from medicinal and aromatic plants*" describes the stages of plant material processing, including harvesting, moisture determination, cutting, and sorting.

  Comparative evaluations of different extraction methods are performed to highlight the influence of parameters such as pressure, time, and ultrasonic power on process efficiency. Hybrid equipment improvement is also analyzed, particularly for systems combining percolation under pressure with ultrasound. The preparation of experimental samples and analysis of the active principles content are carried out using modern analytical techniques to assess the efficiency of extraction conditions.
- **Chapter 6** "*Processing and interpretation of experimental data. Mathematical modeling of the extraction process of active principles from medicinal and aromatic plants*" presents a mathematical model aimed at evaluating the influence of operational parameters on the extraction process. The model allows the prediction of extraction yield and its improvement through surface response analysis and regression equations based on experimental data.
- **Chapter 7** „*General conclusions. Personal contributions. New research directions*", summarizes the results obtained from the theoretical and experimental studies on improving extraction equipment for active principles from medicinal and aromatic plants. The general conclusions highlight the influence of process parameters on the operation of hybrid extraction equipment, the personal contributions brought to the modeling and experimental testing domains, as well as future research directions in the processing, sorting, and extraction of medicinal plants.

CHAPTER 2
CONSIDERATIONS ON MEDICINAL AND AROMATIC PLANTS. ENVIRONMENTAL FACTORS. ACTIVE PRINCIPLES

2.1. Considerations on medicinal and aromatic plants

The use of medicinal and aromatic plants for therapeutic purposes dates back to prehistoric times, and the first written records referring to their use in the treatment of diseases appeared approximately 5000 years ago, marking the beginning of a continuous process of integration and development in medicine (*Hassan, 2015; Jamshidi-Kia et al., 2018; Khan, 2014; Pergola et al., 2024*).

In this context, certain plants stand out due to a unique combination of therapeutic and economic properties. Within this research, attention is focused on three representative species: *nettle* (*Urtica dioica*), *lavender* (*Lavandula angustifolia*) and *sage* (*Salvia officinalis*), which exemplify the potential and complexity of this field. These plants not only reflect the functional diversity of medicinal species but also provide a solid foundation for specific investigations carried out within this study.

- ***Nettle*** (fig. 2.1) is a perennial herbaceous plant belonging to the Urticaceae family, widespread in temperate regions of Europe, Asia, North America, and North Africa (*Bhusal et al., 2022*). From a chemical point of view, nettle is characterized by a diverse bioactive composition, making it a valuable natural resource. The leaves contain polyphenolic compounds such as quercetin and kaempferol, known for their antioxidant and anti-inflammatory properties, as well as phenolic acids (caffeic, ferulic), carotenoids (lutein, beta-carotene), and vitamins (A, C, K, and B complex), minerals such as calcium, magnesium, and potassium.

Fig. 2.1 - Nettle (*Urtica dioica*)

- ***Lavender*** (fig. 2.2) is a perennial aromatic plant belonging to the Lamiaceae family, native to the Mediterranean region. Lavender is well known for its high content of essential oils, among which the most important components are linalool and linalyl acetate. In addition, the plant contains polyphenols, tannins, triterpenes, and phenolic acids, as well as minerals such as potassium, calcium, and magnesium. This complex composition makes lavender an extremely versatile plant (*Prusinowska și Śmigielski, 2014*).

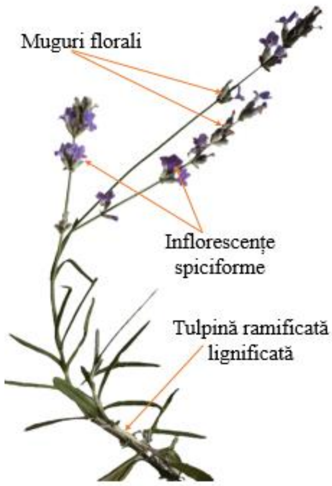


Fig. 2.2 - Lavender (*Lavandula angustifolia*)

- ***Sage*** (fig. 2.3) is a perennial aromatic plant belonging to the Lamiaceae family, native to the Mediterranean region (*Khedher et al., 2017*). From a chemical perspective, sage has a distinctive composition, being rich in essential oils such as thujone and cineole, known for their antimicrobial and antifungal effects. It also contains rosmarinic acid, with strong antioxidant properties, as well as flavonoids with anti-inflammatory effects, and triterpenes and tannins, which have astringent and healing roles. Essential minerals such as calcium, magnesium, iron, and zinc contribute to the nutritional and therapeutic benefits of the plant (*Jassam și Kareem, 2019; Dent et al., 2015; Veličkovic et al., 2006*).

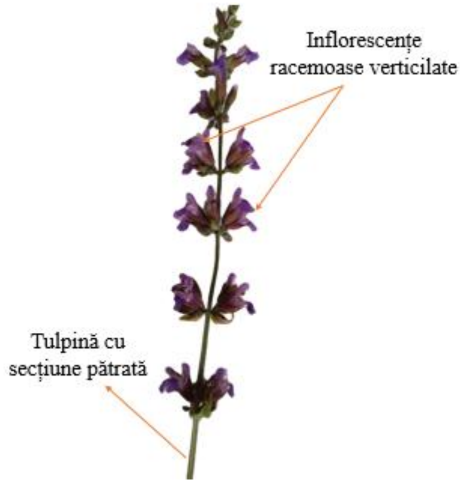


Fig. 2.3 - Sage (*Salvia officinalis*)

Nettle, lavender and *sage* were selected for this research due to their high content of polyphenols, compounds present in significant quantities in the chemical composition of all three species.

2.2. Environmental factors influencing the growth and quality of medicinal and aromatic plants

Climate change and environmental factors such as extreme temperatures, high CO_2 concentrations, ozone levels, soil salinity, light intensity, water quality and availability in the soil, and soil fertility have a complex influence on the development of medicinal plants. These factors cause changes in life cycles, alter secondary metabolism, and drive plant adaptation to stress. This highlights the need for sustainable cultivation and conservation strategies to maintain the quality and productivity of these essential resources (*Pant et al., 2021; Shruti et al., 2024; Laftouhi et al., 2023*).

- ***Temperature***, significantly influences the growth and development of medicinal plants, directly affecting physiological and phenological processes. Elevated temperatures associated with climate change accelerate the stages of plant development, promote earlier flowering, and disturb the balance between primary and secondary metabolic processes (*Shruti et al., 2024; Kalariya et al., 2023; Vâtcă et al., 2020)*.
- ***Carbon dioxide (CO_2) -*** concentration increase in the atmosphere has a significant impact on medicinal and aromatic plants, affecting both growth and phytochemical composition. A higher CO_2 level stimulates photosynthesis, leading to faster growth and greater biomass accumulation. This phenomenon can result in higher leaf, flower, or root yields (*Sharma et al., 2020; Jamloki et al., 2021*).
- ***Tropospheric ozone***, although recognized for its protective effects against UV radiation, induces physiological stress in plants by activating metabolic pathways responsible for the synthesis of bioactive secondary compounds. This phenomenon can be beneficial for some plant species, increasing the concentration of antioxidants and phytochemicals with therapeutic potential, such as phenols and flavonoids. However, ozone can also negatively affect the quality and quantity of metabolites depending on the species' sensitivity and environmental conditions (*Hounsou et al., 2024 ; Madheshiya et al., 2023 ; Han et al., 2023 ; Pradhan et al., 2017)*.
- ***Soil salinity -*** represents a major stress factor for plants, negatively affecting germination, growth, and productivity through nutritional imbalances, hyperosmotic stress, and ionic toxicity, which in turn impact photosynthesis and biochemical processes (*Said-Al Ahl și Omer, 2011; Al Otaibi et al., 2024*).

2.3. Active principles from medicinal and aromatic plants

Medicinal and aromatic plants represent a remarkable source of active principles, used since ancient times for various purposes, ranging from natural remedies to food preservatives.

- ✓ ***Essential oils*** are volatile substances extracted from aromatic plants, used for centuries for their therapeutic and medicinal properties. These compounds are produced in the secondary metabolism of plants and contain a wide range of active substances such as monoterpenes, sesquiterpenes, and phenylpropanoids, which are responsible for their fragrance, properties, and therapeutic efficacy (*de Oliveira et al., 2018; Mustapa et al., 2023)*.
- ✓ ***Polyphenols*** are active principles produced by plants through secondary metabolic processes, characterized by a chemical structure containing an aromatic nucleus and one or more hydroxyl groups, which give them great diversity. Their main classification includes two major categories: flavonoids (compounds such as flavanols, flavonols, anthocyanins, and isoflavones) and non-flavonoids (such as phenolic acids, stilbenes, and lignans). These compounds are commonly found in foods such as fruits, vegetables, cereals, green tea, and also in medicinal and aromatic plants *(Pinto et al., 2021; Mueed et al., 2023; Singla et al., 2019*).
- ✓ ***Alkaloids*** are naturally occurring organic compounds synthesized in the secondary metabolism of plants and occasionally found in fungi, bacteria, and animals. They are defined by the presence of at least one nitrogen atom with basic properties, which gives them a wide range of biological activities and applications. Their diverse structures and complex functions place them among the most studied compounds in the pharmacological and agricultural fields (*El-Saadony et al., 2025)*.
- ✓ ***Saponins*** are glycosidic natural compounds found in many plant species, recognized for their surfactant properties, consisting of a sugar chain bound to a hydrophobic aglycone or sapogenin (*Maghsoudloo et al., 2023; Sharma et al., 2023)*.

CHAPTER 3
EXTRACTION TECHNOLOGIES AND EQUIPMENT FOR OBTAINING ACTIVE PRINCIPLES FROM MEDICINAL AND AROMATIC PLANTS

The extraction of active principles from medicinal and aromatic plants is a complex process that involves the use of various technologies and equipment, adapted according to the type of compound targeted, its sensitivity, and the final application.

3.1. Classical (traditional) methods for extracting active principles from medicinal and aromatic plants

3.1.1. Maceration is a traditional extraction method used to separate active compounds from plant materials by immersing the plants in a solvent without applying direct heat (*Tambun et al., 2021; Hidayat și Wulandari, 2021)*.

3.1.2. Steam distillation is the most widely used method for extracting essential oils, being appreciated for its simplicity and low cost. The process uses steam to release volatile compounds from the plant material, which are then condensed and separated as essential oils (*Machado et al., 2022; Kapadia et al., 2022; Lesage-Meessen et al., 2015)*.

3.1.3. Soxhlet extraction is a continuous extraction method for separating bioactive compounds based on a repetitive cycle of solvent vaporization and condensation. The milled plant material is exposed to a continuous flow of hot volatile solvent, which allows the dissolution of target substances in a repetitive process (*Abubakar și Haque, 2020; Bitwell et al., 2023)*.

3.1.4. Percolation consists of the controlled passage of a solvent through finely ground plant material placed in a special device called a percolator. This method aims to dissolve and extract soluble active principles. Percolation can be performed in two ways:

a) without pressure, using gravity to allow the solvent to flow slowly through the plant layer (*Kumar et al., 2023*).

b) under pressure, where the application of piston-generated pressure enhances solvent penetration into the plant matrix, increasing mass transfer efficiency and extraction yield.

3.2. Classical equipment for the extraction of active principles from medicinal and aromatic plants

3.2.1. Equipment for steam distillation extraction

a) Steam distillation installation IDP500, developed by INMA Bucharest, is designed for the extraction of essential oils from medicinal and aromatic plants and is used both in research centers and by farmers *(Fișă tehnică produs INMA București).*

Fig. 3.1 - Steam distillation installation for medicinal and aromatic plants, IDP500
1 – steam generator, 2 – distillation vessel, 3 – essential oil vapor condenser, 4 – oil separator, 5 – water tanks

3.2.2. Equipment for Soxhlet extraction

A wide range of Soxhlet extractors - from compact 30 mL models, capable of processing between 1 and 8 samples simultaneously, to high-capacity 5000 mL extractors, designed for a single sample - are made by the company Behr Labor-Technik (Germany) (*https://behr-labor.com/en/index.html*).

Fig. 3.2 - Soxhlet extraction equipment KEX 500F/TK (*https://behr-labor.com/en/index.html*)

3.2.3. Equipment for percolation extraction

a) without pressure

There are percolation extraction systems operating without pressure, designed both for laboratory use and industrial production. Figure 3.3 shows a vertical percolator extractor manufactured by ABLE Engineering (India), used for extracting bioactive compounds from medicinal plants through the passage of a liquid solvent through the plant material (*https://www.ableengineers.in/*).

Fig. 3.3 - Percolator-type extractor – ABLE Engineering *(https://www.ableengineers.in/)*

b) under pressure

For pressure percolation extraction, TIMATIC equipment, manufactured by TECNOLAB (Italy), is known for its efficiency and is widely used in the food, pharmaceutical, and plant extract production industries.

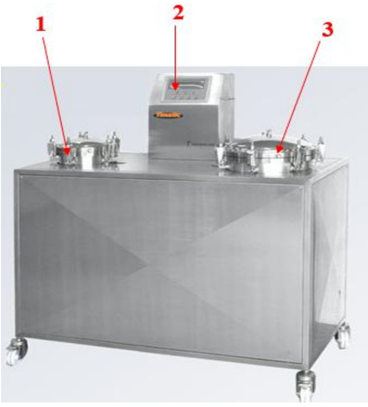


Fig. 3.4 - Duo TIMATIC extractor – TECNOLAB (*www.timatic.it*)

3.3. Modern methods for the extraction of active principles from medicinal and aromatic plants

3.3.1. Microwave-Assisted Extraction (MAE) uses microwave energy to rapidly heat the solvent and plant material, causing cell wall rupture under the combined effect of pressure and high temperature (*Usman et al., 2023; Tsevdou et al., 2024*).

3.3.2. Supercritical Fluid Extraction (SFE) is an environmentally friendly technology that uses carbon dioxide (CO_2) in its supercritical state to extract active compounds from various materials. The method involves compressing and heating CO_2 to bring it into a supercritical state, followed by passing it through the solid plant material for compound extraction (*Tăbărașu et al.,2023*).

3.3.3. Ultrasound-Assisted Extraction (UAE) is a modern and efficient technology for isolating active principles from plant materials, optimizing the process by reducing time and resource consumption. In UAE, sound waves propagate through the liquid as a series of compression and rarefaction cycles, generating intense vibrations that trigger the phenomenon of cavitation (*Shen et al., 2023)*.

3.4. Modern equipment for the extraction of active principles from medicinal and aromatic plants

3.4.1. Microwave extraction equipment

Among the microwave extraction systems, the Milestone EOS-G model is notable for laboratory use, while for pilot-scale processing, the MAC-75 equipment, from the study by

(*Petigny et al., 2014*), both devices being used for the extraction of organic compounds from plant leaf material.

a)

b)

Fig. 3.5 - Microwave extraction equipment (*Petigny et al., 2014*)
a) Milestone EOS-G: 1 – condenser, 2 – essential oil, 3 – cohobation system, 4 – aqueous phase, 5 – microwave oven, 6 – microwave reactor, 7 – plant material;
b) MAC-75: 1 – microwave reactor, 2 – condenser.

3.4.2. Ultrasound extraction equipment

There is a wide range of ultrasound extraction systems adapted for different applications, from laboratory models with a power of 50 W to large-capacity industrial equipment reaching up to 16,000 W.

Fig. 3.6 - Ultrasonicator UP400St
(*https://www.hielscher.com*)

Fig. 3.7 - Sonicator UIP16000
(*https://www.hielscher.com*)

CHAPTER 4
CURRENT STATE OF RESEARCH ON THE EXTRACTION PROCESS OF ACTIVE PRINCIPLES FROM MEDICINAL AND AROMATIC PLANTS

4.1. Current state of theoretical research on the extraction process of active principles from medicinal and aromatic plants

To optimize ultrasound-assisted extraction of active principles from plant materials, various statistical methods are used to help understand the relationship between factors and the final result.

- *Response Surface Methodology (RSM)* is frequently used as a tool for optimizing technological processes, including the extraction of active principles from medicinal and aromatic plants. From a mathematical perspective, RSM is based on predictive models, usually expressed as second-order polynomial equations (Equation 4.1), which allow the estimation of the behavior of the studied system (*Anaya-Esparza et al., 2023*).

$$Y = b_0+b_1X_1+b_2X_2+b_3X_3+b_{11}X_1^2+b_{22}X_2^2+b_{33}X_3^2+b_{12}X_1X_2+b_{13}X_1X_3+b_{23}X_2X_3 \quad (4.1)$$

where:

Y – predicted response value;
X – independent factor;
b_1, b_2, b_3 – regression coefficients for the linear effect;
b_{11}, b_{22}, b_{33} – coefficients for the quadratic effect;
b_{12}, b_{13}, b_{23} – coefficients for the linear interaction effect.

- *Fractional Factorial Design* - analyzes only a portion of all possible combinations while maintaining a representative model of the process. It is especially useful in the early stages of research to eliminate nonessential factors before detailed optimization. Such a design is expressed as 2^{k-m}.

where:

k - total number of factors considered,
m - number of factors excluded to reduce the number of experiments (*Anaya-Esparza et al., 2023*).

- *Plackett–Burman Design (PBD)* - selects only the most significant factors for the process, saving resources and time. The equation used for this design is simplified as follows:

$$Y = \beta_0 + \sum \beta_i X_i \quad (4.2)$$

where the coefficient β_i with the highest absolute value indicates the factor that most strongly influences the process (*Anaya-Esparza et al., 2023*).

4.2. Current state of experimental research on the extraction process of active principles from medicinal and aromatic plants

- A representative example of the application of the Response Surface Methodology (RSM) is provided by the study (*Muzykiewicz-Szymańska et al., 2024*) which aimed to optimize ultrasound-assisted extraction of bioactive compounds from the aerial parts of *Sanguisorba officinalis* L. The process was conducted in an ultrasonic bath (40 kHz, 40 ± 1 °C) using ground plant material with particle size below 0.25 mm. The parameters included in the experimental design were the concentration of plant material (2.25–7.5 g/100 mL), ethanol concentration (20–60%), and extraction time (1–15 minutes). The response surface modeling was performed using a second-order polynomial equation, as expressed below:

$$Y_i = a_0 + \sum_{i=1}^{3} a_{i1} X_i + \sum_{i=1}^{3} a_{i2} {X_i}^2 + \sum_{i=1}^{2} \sum_{j=i+1}^{3} a_{ij} X_i X_j \tag{4.3}$$

where: X_i represents the independent factors of the process (raw material content, ethanol concentration, extraction time), a_i are the regression coefficients, and Y_i is the dependent variable, namely the antioxidant activity and compound content.

The optimal parameters determined through RSM were an extraction time of 10 minutes, ethanol concentration of 47% v/v, and raw material content of 7.5 g/100 mL. Under these conditions, the highest measured values were obtained according to the proposed objectives. The graphical results (fig. 4.1, fig. 4.2) illustrate the combined effects of the process parameters on compound content and antioxidant activity, highlighting the relationship between process conditions and extraction efficiency.

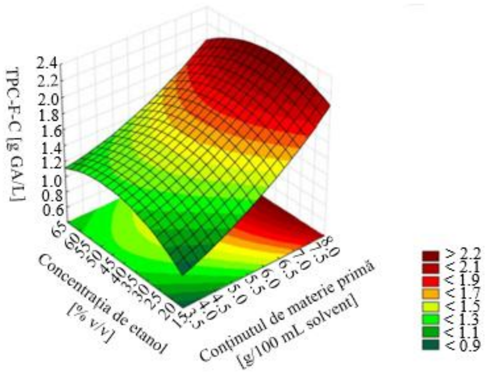


Fig. 4.1 - Effect of raw material content and ethanol concentration on the bioactive compound content; extraction time: 10 minutes (*Muzykiewicz-Szymańska et al., 2024*)

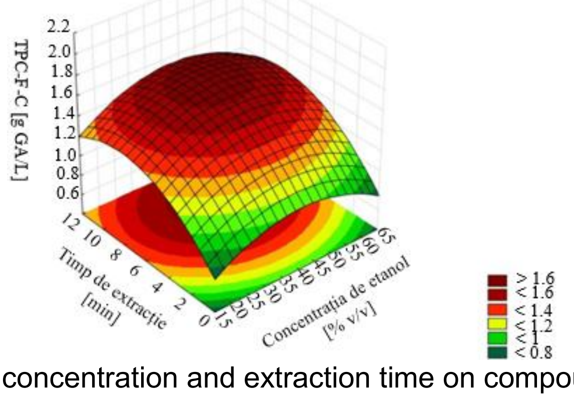


Fig. 4.2 - Effect of ethanol concentration and extraction time on compound content; raw material: 7.5 g/100 mL (*Muzykiewicz-Szymańska et al., 2024*)

CHAPTER 5
EXPERIMENTAL RESEARCH ON IMPROVING EXTRACTION EQUIPMENT PERFORMANCE FOR ACTIVE PRINCIPLES FROM MEDICINAL AND AROMATIC PLANTS

The objectives of the experimental research aimed at improving hybrid extraction equipment for pressure percolation–ultrasound extraction used in the processing of medicinal and aromatic plants are:

- developing a general experimental research methodology for obtaining concentrated plant extracts using hybrid extraction technologies;
- selecting and configuring the equipment used in the experimental process to ensure compatibility with the technological requirements of hybrid extraction systems;
- determining the moisture content of dried medicinal plants, followed by testing the grinding process performance on different particle sizes of plant material to select the optimal particle size for the extraction stage;
- obtaining plant extracts using a common solvent, applied to three different plant species: nettle, lavender, and sage;
- evaluating the influence of plant species on extraction yield to identify the variant that ensures the best balance between extraction efficiency and energy consumption associated with the grinding of medicinal plants;
- determining the maximum concentrations of active principles (polyphenols) in plant extracts (through UV-VIS absorption spectra analysis) to assess extract quality and compositional accuracy.

5.1. Processing of plant material and selection of extraction method

5.1.1. Characterization and preparation of plant material (harvesting, moisture determination, plant characterization)

a) b) c)

Fig. 5.1 - Medicinal and aromatic plant crops:
a) nettle; b) lavender; c) sage

The plant material used in this study was obtained from the harvesting of three species of medicinal and aromatic plants: nettle, lavender, and sage (fig. 5.1).

Nettle plants were harvested manually, at a distance of approximately 5 cm from the ground. Lavender and sage plants were harvested mechanically using specialized equipment designed for collecting medicinal and aromatic plants, which ensures a uniform cut of the plant material. The cutting height was adjusted to approximately 10–15 cm above ground level for sage and about 15 cm for lavender.

In this study, the plant material was subjected to a natural drying process, carried out on-site, at an ambient temperature of approximately 25°C. The moisture content of the three plants studied: nettle, lavender, and sage, decreased from about 80% (nettle), 55% (sage), and 50% (lavender) after 14 days to approximately 10% (fig. 5.2).

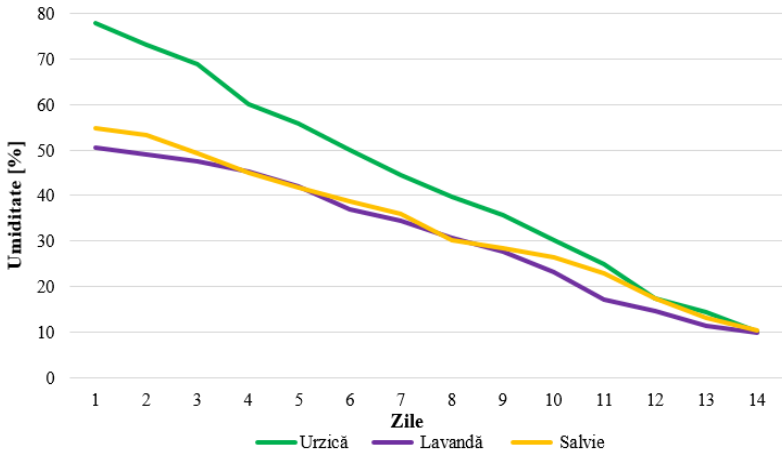


Fig. 5.2 - Variation of moisture content during the drying process (14 days) for: nettle, lavender, and sage

The preliminary evaluation also included the determination of active principles content. To estimate the initial content, samples of dried plants were treated with water and kept at rest for approximately 24 hours at room temperature under non-forced conditions. This stage did not represent a standardized extraction method and had no technological purpose, serving solely an informative role aimed at obtaining an approximate estimation of the active principles present in the analyzed plants prior to the application of the hybrid extraction method. The total content of active principles was determined from the extract using the Folin–Ciocalteu reagent, and the results were expressed as active compound content (mg GAE/100 g) (tabel 5.1).

Table 5.1

Initial content of active compounds identified in the studied plants

Active principles content, *Cp*, for nettle [mg GAE/100g]	**Active principles content, *Cp*, for lavender [mg GAE/100g]**	**Active principles content, *Cp*, for sage [mg GAE/100g]**
14.97	28.03	33.18

5.1.2. Cutting of plants for the extraction method selection stage

The processing of plant material continued with mechanical cutting performed using the *HERBCUT TIMATIC* equipment (Tecnolab srl, Spello, Italy), designed to ensure efficient fragmentation of medicinal and aromatic plants (fig. 5.3). Initially, a portion of the plants was cut to a size of 30 mm, later used for testing three extraction methods: pressure percolation, ultrasound extraction, and the hybrid method of pressure percolation assisted by ultrasound.

Fig. 5.3 - Aspects during the mechanical cutting of medicinal and aromatic plants using HERBCUT TIMATIC

The HERBCUT equipment allows obtaining variable cutting sizes depending on subsequent processing requirements, being characterized by a robust construction, energy efficiency, and adaptability to different types of plant material.

5.1.3. Selection of the most efficient extraction method

To identify the most efficient extraction method, five extraction techniques were investigated, ranging from classical percolation methods at low and high pressure to modern combinations that include ultrasonic treatment. All samples were cut to a size of 30 mm, with a plant material amount of 400 g per sample, and water was used as the extraction solvent. Experimental results showed that combining high-pressure percolation with ultrasonic treatment provides the highest yields in terms of extracting bioactive compounds (*Cp*) for all three plant species analyzed in this study.

Tables 5.2−5.4 present the process parameters used for each method and the values obtained following their application.

Table 5.2

Comparison of extraction methods for nettle based on the influence of process parameters, using active principles content as an efficiency indicator

Extraction method	Pressure [bar]	Time [min]	Ultrasound power [W]	Active principles content, *Cp*, for nettle [mg GAE/100g]
Low-pressure percolation	5	60	-	*5.19*
High-pressure percolation	7	60	-	*6.24*
Low-pressure percolation assisted by ultrasound	5	60	140	*7.58*
High-pressure percolation assisted by ultrasound	7	60	140	*8.16*
Ultrasound-assisted extraction	-	60	140	*7.09*

The extraction methods were also tested for longer processing times:90, 120, and 150 minutes and based on the results, only three extraction time values were selected: 60, 90, and 120 minutes. This is because at 150 minutes the active principles content increased by only 1.21% compared to 120 minutes (t > 25%), a result considered insignificant in terms of yield.

Table 5.3

Comparison of extraction methods for lavender based on the influence of process parameters, using active compound content as an efficiency indicator

Extraction method	Pressure [bar]	Time [min]	Ultrasound power [W]	Active principles content, *Cp*, for lavender [mg GAE/100g]
Low-pressure percolation	5	60	-	*14.46*
High-pressure percolation	7	60	-	*15.07*
Low-pressure percolation assisted by ultrasound	5	60	140	*15.13*
High-pressure percolation assisted by ultrasound	7	60	140	*16.90*
Ultrasound-assisted extraction	-	60	140	*13.52*

Table 5.4

Comparison of extraction methods for sage based on the influence of process parameters, using active compound content as an efficiency indicator

Extraction method	Pressure [bar]	Time [min]	Ultrasound power [W]	Active principles content, *Cp*, for sage [mg GAE/100g]
Low-pressure percolation	5	60	-	*17.19*
High-pressure percolation	7	60	-	*20.75*
Low-pressure percolation assisted by ultrasound	5	60	140	*23.10*
High-pressure percolation assisted by ultrasound	7	60	140	*24.24*
Ultrasound-assisted extraction	-	60	140	*21.51*

Based on the values in Tables 5.6–5.8, it is observed that ultrasound-assisted high-pressure percolation is the most efficient extraction method, providing higher yields than the other methods analyzed.

5.1.4. Evaluation of the influence of cutting size for the hybrid method of pressure percolation assisted by ultrasound

To determine the optimal cutting size of the plant material intended for extraction, preliminary tests were performed using three different fragmentation sizes: 30 mm / 16 mm / 12 mm. After preliminary cutting with the Herb*Cut* equipment, the cut plant material was transferred by the inclined belt conveyor (fig. 5.4).

Fig. 5.4 - Inclined belt conveyor:
1 – upper drum, 2 – support frame, 3 – conveyor belt, 4 – lower drum

The next stage consisted of sorting the plant material using the cut plant sorter (fig. 5.5), a specialized device designed to separate plant fragments into three distinct size fractions, depending on the mesh size of the sieves used.

Fig. 5.5 - Cut medicinal and aromatic plant sorter*:*
1 – feed hopper for plant material, 2 – sieve frames, 3 – outlets for sorted plant fractions, 4 – electrovibrating motor, 5 – sieve support, 6 – support frame, 7 – vibration dampers

After completing the preparatory stages of the plant materials, the preliminary extraction experiments were carried out, with the objective of identifying the method with the highest efficiency. These were performed under constant processing conditions: extraction time of 60 minutes, percolation pressure of 7 bar, ultrasound power of 100 W, and water was used as the solvent. The results obtained, presented in tables 5.5 to 5.7, reflect the combined influence of the degree of grinding 12 / 16 / 30 mm and the amount of plant material 200 / 400 / 600 g on the extraction process, with the content of active principles as the indicator.

Table 5.5

Effect of cutting size and plant material quantity on the extraction yield of active principles from *nettle*

Pressure [bar]	Time [min]	Ultrasound power [W]	Plant material quantity [g]	Cutting size [mm]	Active principles content, *Cp*, for nettle [mgGAE/100g]
7	60	100	200	30	5.57
7	60	100	200	16	6.02
7	60	100	200	12	6.23
7	60	100	400	30	8.87
7	60	100	400	16	9.47
7	60	100	400	12	10.43
7	60	100	600	30	10.05
7	60	100	600	16	11.08
7	60	100	600	12	11.55

Table 5.6

Effect of cutting size and plant material quantity on the extraction yield of active principles from *lavender*

Pressure [bar]	Time [min]	Ultrasound power [W]	Plant material quantity [g]	Cutting size [mm]	Active principles content, Cp, for lavender [mgGAE/100g]
7	60	100	200	30	11.53
7	60	100	200	16	12.39
7	60	100	200	12	12.89
7	60	100	400	30	14.93
7	60	100	400	16	15.89
7	60	100	400	12	16.99
7	60	100	600	30	16.32
7	60	100	600	16	17.96
7	60	100	600	12	18.47

Table 5.7

Effect of cutting size and plant material quantity on the extraction yield of active principles from *sage*

Pressure [bar]	Time [min]	Ultrasound power [W]	Plant material quantity [g]	Cutting size [mm]	Active principles content, Cp, for sage [mgGAE/100g]
7	60	100	200	30	23.41
7	60	100	200	16	25.26
7	60	100	200	12	25.97
7	60	100	400	30	26.81
7	60	100	400	16	28.50
7	60	100	400	12	30.10
7	60	100	600	30	27.47
7	60	100	600	16	30.20
7	60	100	600	12	30.27

The analysis of the values from tables 5.5–5.7 shows that the variations resulting from the cutting size and plant material quantity are not very large, with percentage differences below 20%. Reducing the fragment size from 30 mm to 16 mm and 12 mm did not produce significant changes in the overall extraction efficiency, according to the determined values.

In addition, for nettle, the energy consumption required for cutting a larger quantity of 25 kg was 0.109 kWh at a cutting size of 30 mm. At 16 mm, the consumption increased by 25% (0.136 kWh), and at 12 mm by 31% (0.143 kWh).

For lavender, an energy consumption of 0.132 kWh was recorded at a cutting size of 30 mm, while the values increased by 28% at 16 mm (0.169 kWh) and by 35% at 12 mm (0.178 kWh).

For sage, the energy consumption for cutting the same amount of material was 0.156 kWh at a cutting size of 30 mm, increasing by 30% at 16 mm (0.203 kWh) and by 38% at 12 mm (0.215 kWh).

Reducing the fragment size, in all cases, requires a longer cutting effort, which results in higher load on the drive system and the cutting assembly. The higher frequency of cuts and prolonged contact with the plant material, especially for species with dense or woody structures, can accelerate blade wear and increase the load transmitted to the equipment motor.

Consequently, considering the need to optimize energy consumption during the cutting process and to improve the efficiency of the technological process, a cutting size of ***30 mm*** was selected for the subsequent stages of the study, while maintaining a constant plant material quantity of ***400 g***. This choice ensures an effective balance between extraction efficiency and the energy costs associated with plant material preparation.

5.2. Selection of the optimal ultrasound extraction power

Before the actual experiments, additional tests were performed in which the ultrasound power was varied at 80, 100, 120, 140, and 160 W to evaluate the influence of this parameter on the efficiency of the extraction process. The cutting size of the plants used in the experiments was 30 mm, and the plant material quantity was 400 g. The results indicated the power level at which the transfer of active principles from the plant material occurred most efficiently (tables 5.8–5.10).

Table 5.8

Effect of ultrasound power variation on extraction efficiency for nettle

Pressure [bar]	Time [min]	Ultrasound power [W]	Active compound content, *Cp*, for nettle [mg GAE/100g]
5	60	80	7.53
5	60	100	8.16
5	60	120	9.28
5	60	140	7.58
5	60	160	5.71
6	60	80	7.82
6	60	100	8.65

6	60	120	9.77
6	60	140	7.63
6	60	160	5.90
7	60	80	7.95
7	60	100	8.87
7	60	120	10.88
7	60	140	8.16
7	60	160	6.25

For nettle, the experiments showed that applying an ultrasound power of 120 W, under constant extraction time (60 minutes) and variable pressures (5, 6, 7 bar), led to the highest process efficiency in terms of extraction yield. Exceeding this power threshold resulted in a decrease in process performance, most likely due to intensified cavitation, which can negatively affect process stability through excessive mechanical effects on the plant matrix.

Table 5.9

Effect of ultrasound power variation on extraction efficiency for lavender

Pressure [bar]	Time [min]	Ultrasound power [W]	Active compound content, Cp, for lavender [mg GAE/100g]
5	60	80	15.10
5	60	100	14.40
5	60	120	15.16
5	60	140	15.13
5	60	160	14.73
6	60	80	16.25
6	60	100	14.58
6	60	120	16.60
6	60	140	16.09
6	60	160	15.85
7	60	80	16.39
7	60	100	14.93
7	60	120	16.92
7	60	140	16.89
7	60	160	16.02

For lavender, the tests conducted at 60 minutes, pressures between 5 and 7 bar, and ultrasound powers ranging from 80 to 160 W indicated that the highest extraction efficiency was achieved at a pressure of 7 bar and an acoustic power of 120 W. At higher power levels (140–160 W), the efficiency slightly decreased, suggesting that beyond a certain energy threshold, cavitation effects become less favorable for the process, probably due to excessive stress on the plant structure and disturbance of the extraction equilibrium.

Table 5.10

Effect of ultrasound power variation on extraction efficiency for sage

Pressure [bar]	Time [min]	Ultrasound power [W]	Active compound content, Cp, for sage [mg GAE/100g]
5	60	80	20.04
5	60	100	25.55
5	60	120	23.29
5	60	140	23.10
5	60	160	22.85
6	60	80	22.59
6	60	100	26.79
6	60	120	23.81
6	60	140	23.21
6	60	160	22.61
7	60	80	24.57
7	60	100	26.81
7	60	120	24.40
7	60	140	24.24
7	60	160	23.90

For sage, the tests conducted at a constant time of 60 minutes, pressures between 5 and 7 bar, and ultrasound powers ranging from 80 to 160 W showed that the maximum extraction efficiency was obtained at a pressure of 6 bar and an acoustic power of 100 W. Subsequently, as the power increased to 120 W and above, lower values were recorded, indicating a decreasing trend. However, at a pressure of 7 bar, the values began to increase again at higher intensities, without reaching the previous level, suggesting an oscillating response of the system in relation to ultrasound power, possibly influenced by the structural characteristics of the plant material.

5.3. Evaluation of the efficiency of hybrid extraction equipment for pressure percolation assisted by ultrasound based on active principles content

The experimental installation consists of a TIMATIC Duo extractor (Tecnolab srl, Spello, Italy) modified with an ultrasound generator UP400St (Hielscher Ultrasonics GmbH, Teltow, Germany), configured to allow simultaneous operation of two processes using the same extraction method.

The TIMATIC Duo extractor (fig. 5.6) is an automated system designed for solid–liquid extraction under controlled pressure, using compatible liquids such as water, ethanol, or other solvents. In its original factory configuration, the system operates within a pressure range of 3 to 7 bar and is equipped with two extraction chambers with active volumes of 12 and 24 liters, respectively. The equipment includes two hydropneumatic cylinders, a pump, solenoid valves for flow control, a solvent reservoir, a pneumatic distributor, and a pressure sensor for real-time monitoring of process parameters.

To adapt the equipment to the specific requirements of the experimental application, the original configuration was modified by removing the 24-liter extraction chamber, maintaining the 12-liter chamber. The chamber is made of stainless steel, a material chosen for its corrosion resistance and compatibility with a wide range of solvents used in extraction processes. The chamber allows the introduction of plant material packed into permeable mesh bags, ensuring easy handling and a high degree of process repeatability.

Fig. 5.6 - TIMATIC Duo extraction equipment:
1 – control panel, 2 – extraction chamber, 3 – support frame, 4 – solvent tank, 5 – compressor, 6 – extract collection vessel

To enable the implementation of hybrid extraction, the extraction vessel of the percolator was technically modified by integrating an ultrasonic probe directly inside the extraction chamber (figs. 5.7 and 5.8). The probe is connected to the UP400St system, a laboratory ultrasonic generator operating at a fixed frequency of 24 kHz and adjustable power between 0 and 400 W. The equipment allows the adjustment of ultrasonic vibration amplitude within a range of 20 to 100%, depending on the process requirements. The probe used is made of titanium, with a tip diameter of 19 mm and a length of approximately 215 mm. The ultrasonic energy generated by this system produces cavitation phenomena in the liquid medium, forming microbubbles that violently implode near the cell walls of the plant material. This mechanical action promotes the disruption of cellular structures and the rapid release of intracellular compounds, resulting in a significant increase in extraction efficiency without requiring additional temperature increase or the use of aggressive solvents.

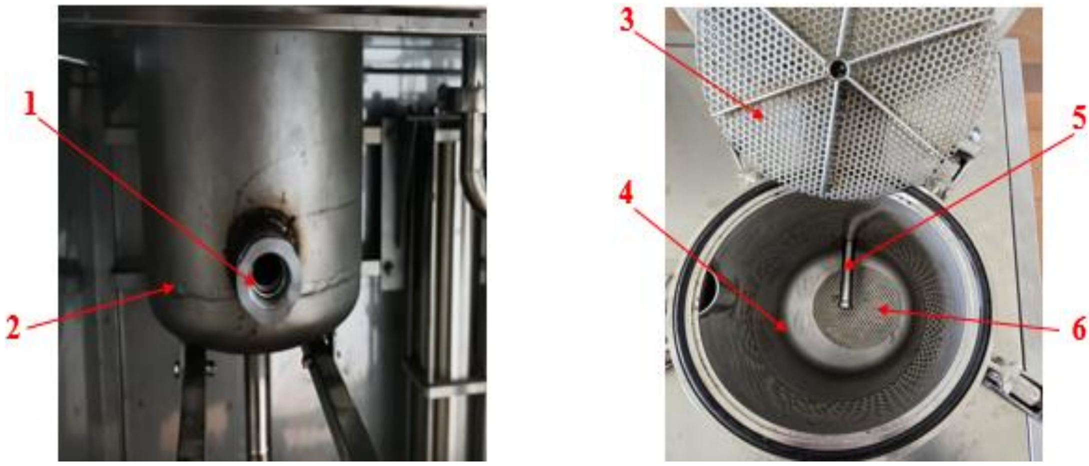


Fig. 5.7 - Integration of the Hielscher UP400St sonicator into the extraction chamber of the TIMATIC Duo percolator: *1 – ultrasonic probe insertion port; 2, 4 – extraction chamber; 3 – upper sieve; 5 – sonotrode; 6 – lower sieve*

Fig. 5.8 - Aspects during the extraction process:
1 – extraction chamber, 2 – sonotrode, 3 – ultrasonic generator and control unit (Hielscher UP400St)

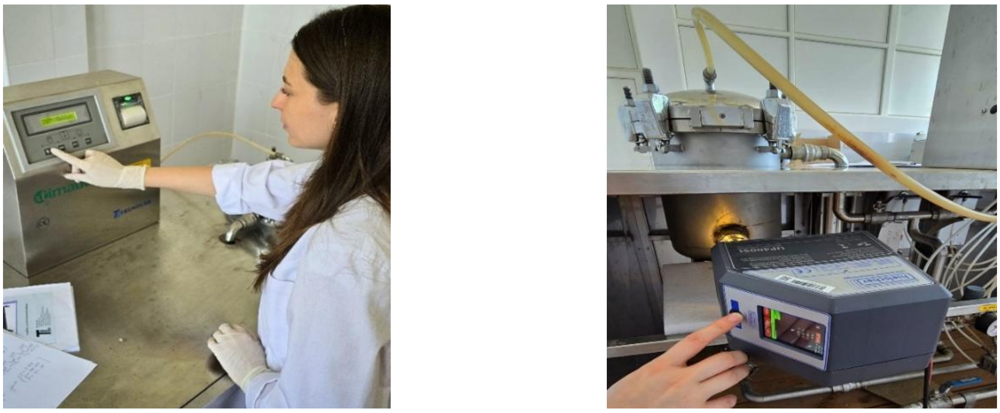
5.3.1. Preparation of plant samples for experimental tests

After the preliminary preparation of the plant material, drying, cutting and granulometric sorting, individual samples of 400 g were weighed and placed into permeable mesh bags, being thus prepared for the extraction of active principles.

The bags were positioned inside the extraction chamber, over which 12 liters of water were added (fig. 5.9). For the system to operate correctly, the installation automatically extracts approximately 7 liters of buffer solvent from the reservoir, this quantity not coming into contact with the sample but being used to complete the internal circuit and maintain the pressure required during the process. Water was used as the extraction solvent because of its mild character and compatibility with the structure of the plant material, as well as because it does not promote the degradation of sensitive active principles.

Fig. 5.9 - Aspects during the preparation of samples for the extraction process

After loading the extraction chamber and filling the circuit with solvent, the operating parameters were set using the digital control panel of the percolation unit and the touchscreen display of the ultrasonic generator (fig. 5.10).



(a) (b)

Fig. 5.10 - Adjustment of input parameters at*:*
(a) digital control panel of the percolator, (b) touchscreen display of the ultrasonic generator

In the experimental study, three operating parameters were varied in a controlled manner: working pressure, extraction duration, and applied ultrasound power, in order to evaluate their impact on extraction yield, expressed as active principles content (tabel 5.11).

Table 5.11

Parameters of the hybrid extraction process: pressure percolation assisted by ultrasound

Pressure [bar]	Time [min]	Ultrasound power [W]
5	60	80
6	60	80
7	60	80
5	60	100
6	60	100
7	60	100
5	60	120
6	60	120

7	60	120
5	90	80
6	90	80
7	90	80
5	90	100
6	90	100
7	90	100
5	90	120
6	90	120
7	90	120
5	120	80
6	120	80
7	120	80
5	120	100
6	120	100
7	120	100
5	120	120
6	120	120
7	120	120

A particular feature of the applied extraction method lies not only in the combination of pressure percolation with ultrasound but also in the way pressure is applied, performed cyclically through the alternation of two phases, static and dynamic (fig. 5.11). During the extractions, this cyclic pressure percolation allowed efficient control of the process, each cycle contributing to improved contact between the solvent and the plant material.

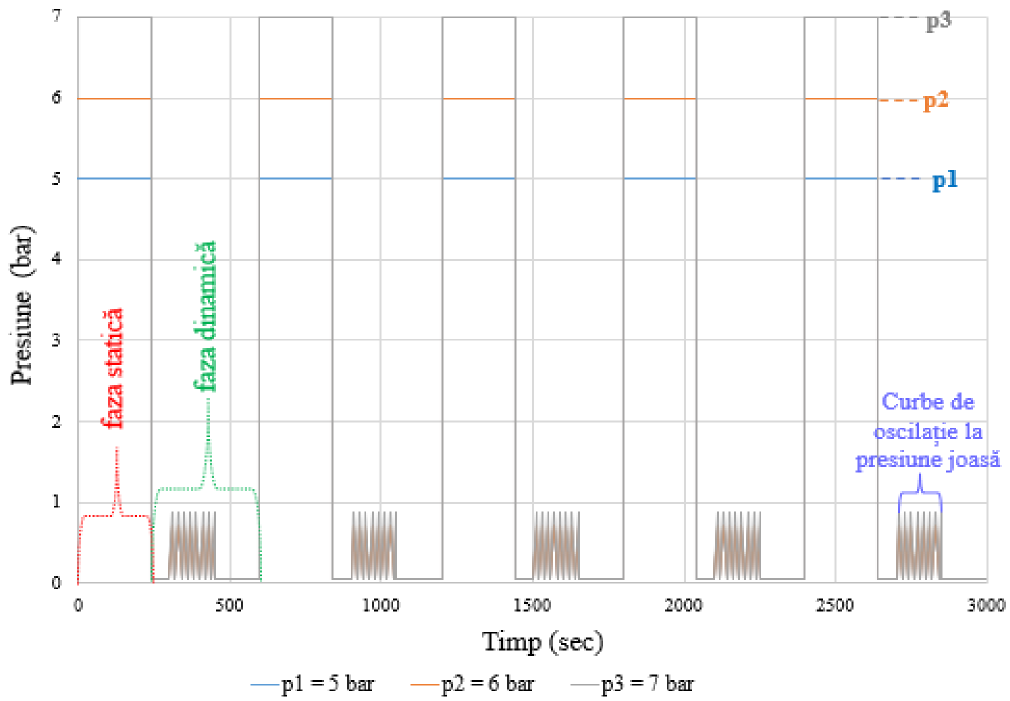


Fig. 5.11 - General diagram of extraction pressures in the process: static phase and dynamic phase

- Static phase (the first 240 seconds of each cycle) consists of applying a constant pressure of 5, 6, or 7 bar, depending on the experimental conditions, without variation.
- Dynamic phase (the following 360 seconds) is characterized by cyclic oscillations of the pressure between a high and a low value, for example: between 0.69 and 0.06 bar for the 5-bar pressure, and between 0.87 and 0.12 bar for the 7-bar pressure.

The alternation between the two phases is repeated throughout the entire extraction period: 6 cycles for 60 minutes, 9 cycles for 90 minutes, and 12 cycles for 120 minutes. In each cycle, the static (compression) phase has a constant duration of 4 minutes, while the dynamic (decompression) phase lasts for 6 minutes.

5.3.2. Validation of the extraction method according to the content of resulting active principles

The extraction efficiency was verified using the Folin–Ciocalteu method by measuring absorbance at 755 nm and comparing the results with the calibration curve for gallic acid, expressed in [mg GAE/g] or [mg GAE/100 g]. For each combination of extraction parameters, values were obtained that allow the evaluation of their influence on the process. The results are presented in tables 5.12–5.14, in accordance with the experimental conditions described in table 5.11.

Table 5.12

Correlation between process parameters and extraction yield of active principles from nettle

Pressure [bar]	Time [min]	Ultrasound power [W]	Active principles content, *Cp,* for nettle [mg GAE/100g]
5	60	80	7.53
6	60	80	7.82
7	60	80	7.95
5	60	100	8.16
6	60	100	8.65
7	60	100	8.87
5	60	120	9.28
6	60	120	9.77
7	60	120	10.88
5	90	80	8.56
6	90	80	8.78
7	90	80	9.02
5	90	100	8.33
6	90	100	8.39
7	90	100	9.22
5	90	120	9.43
6	90	120	9.86
7	90	120	10.50
5	120	80	6.59
6	120	80	9.03
7	120	80	12.55
5	120	100	8.10

6	120	100	9.80
7	120	100	12.92
5	120	120	8.64
6	120	120	11.97
7	120	120	13.71

Table 5.13

Correlation between process parameters and extraction yield of active principles from lavender

Pressure [bar]	Time [min]	Ultrasound power [W]	Active principles content, *Cp*, for lavender [mg GAE/100g]
5	60	80	15.10
6	60	80	16.25
7	60	80	16.39
5	60	100	14.40
6	60	100	14.58
7	60	100	14.93
5	60	120	15.16
6	60	120	16.60
7	60	120	16.93
5	90	80	16.95
6	90	80	18.43
7	90	80	20.41
5	90	100	17.49
6	90	100	18.03
7	90	100	18.56
5	90	120	22.95
6	90	120	23.04
7	90	120	23.16
5	120	80	21.99
6	120	80	22.46
7	120	80	23.21
5	120	100	19.71
6	120	100	22.59
7	120	100	24.28
5	120	120	23.12
6	120	120	24.24
7	120	120	25.91

Table 5.14

Correlation between process parameters and extraction yield of active principles from sage

Pressure [bar]	Time [min]	Ultrasound power [W]	Active principles content, *Cp*, for sage [mg GAE/100g]
5	60	80	20.04
6	60	80	22.59
7	60	80	24.57
5	60	100	25.55
6	60	100	26.79
7	60	100	26.81
5	60	120	23.29
6	60	120	23.81

7	60	120	24.40
5	90	80	25.51
6	90	80	25.70
7	90	80	25.83
5	90	100	24.45
6	90	100	24.66
7	90	100	29.35
5	90	120	25.34
6	90	120	26.50
7	90	120	30.40
5	120	80	21.49
6	120	80	24.97
7	120	80	26.93
5	120	100	24.16
6	120	100	26.44
7	120	100	28.72
5	120	120	28.15
6	120	120	28.94
7	120	120	30.79

Based on the experiments performed (tables 5.12–5.14), it was observed that the pressure, duration, and ultrasound power used during the extraction process directly influence the active principles that can be extracted from the analyzed plants.

A higher pressure (7 bar) led to the extraction of a larger amount of active principles (polyphenols). This occurs because the liquid penetrates deeper into the plant material when pushed with greater force, thus extracting the compounds more efficiently. The ultrasound power (used to agitate the mixture and break the plant cell structure) also had a significant effect. The higher the applied ultrasound power (120 W), the better the extraction yield, especially under high pressure. Therefore, the most efficient set of parameters that produced the highest amount of active principles (within the studied range) were:

- pressure: 7 bar;
- extraction time: 120 min;
- ultrasound power: 120 W.

5.3.3. Correlation between energy consumption and extraction process efficiency

To correlate the efficiency of the extraction process with the energy requirement, the energy consumption of the equipment was evaluated under the most efficient parameters: extraction time of 120 minutes, variable pressures of 5, 6, and 7 bar, and ultrasound powers of 80, 100, and 120 W (table 5.15).

Tabel 5.15
Total energy consumption of the hybrid extraction equipment for pressure percolation assisted by ultrasound under the most efficient process parameters

Time [min]	Pressure [bar]	Ultrasound power [W]	Total energy consumption [kWh]
120	5	80	0.450
120	6	80	0.459
120	7	80	0.466
120	5	100	0.473
120	6	100	0.481
120	7	100	0.492
120	5	120	0.507
120	6	120	0.519
120	7	120	0.528

The comparative analysis of efficiency and energy consumption data shows a direct correlation between the intensification of technological parameters (pressure and ultrasound power) and the increase in active principles content. For *nettle*, the highest percentage increase was recorded, approximately 108%, with an energy consumption about 17% higher, suggesting an efficient ratio between yield and energy used.

In the case of *lavender*, the increase in active principles content was about 18%, almost proportional to the 17% increase in energy consumption. This ratio indicates that, although the extraction efficiency increase was more modest compared to the other species, the energy efficiency remained balanced without significant additional consumption.

For *sage*, an increase of approximately 43% in active principles content was obtained with an energy consumption similar to that of nettle (around 17%), confirming the good performance of the equipment and the selected set of operating parameters.

CHAPTER 6
PROCESSING AND INTERPRETATION OF EXPERIMENTAL DATA. MATHEMATICAL MODELING OF THE EXTRACTION PROCESS OF ACTIVE PRINCIPLES FROM MEDICINAL AND AROMATIC PLANTS

6.2. Statistical Modeling

NETTLE

6.2.3. Statistical results of multivariate polynomial regressions

The Multiple Linear Regression Calculator function from the Mathcad 15 software was used to calculate these regressions. In increasing order of polynomial interpolation degrees, the coefficients of the statistical regression polynomials were obtained (table 6.1):

Table 6.1

Statistical regression polynomial coefficients, rejection probabilities, and determination coefficient for the dependent variable, *Cp*

Term	Coeff. Gr. 1	Refusal test	Coeff. Gr. 2	Refusal test
$p^0t^0P^0$	-4.479037	0.0420000	34.194546	0.033431
p	1.166000	0.0001099	-3.886500	0.219219
t	0.026650	0.0042100	-0.275558	0.002799
P	0.045036	0.0015850	-0.166040	0.224380
pt	-	-	0.036453	0.000049
pP	-	-	0.003704	0.366373
tP	-	-	-0.000064	0.385658
p^2	-	-	0.116778	0.360198
t^2	-	-	0.000500	0.116437
P^2	-	-	0.000973	0.155541

The results obtained using the Multiple Linear Regression Calculator program are presented in table 6.1. It was observed that for the complete second-degree polynomial, the calculated probabilities of coefficient rejection were, in most cases, lower than the significance level of 0.05. These values show that polynomial interpolation is possible and reliable, especially for first-degree polynomials. However, there are also terms in the complete second-degree polynomial that are slightly below or just above the 0.05 significance level. These terms were therefore accepted for the second-degree polynomial regression selected by the multivariate regression analysis program PTC Mathcad Prime 9.0.0.0. For the second-degree progression, whose coefficients are presented in table 6.1, the determination coefficient had a value of 0.889, and the adjusted determination coefficient was 0.83, resulting in the nonlinear regression (6.1). For the second-degree polynomial, using the statistical analysis program PTC Mathcad Prime 9, the final formula (6.1) was selected:

$$C_p = 17.371707 - 2.11475p - 0.192067t + 0.0364528pt + 0.000227666P^2 \quad (6.1)$$

The determination coefficient reaches R^2=0,86, and the adjusted determination coefficient is R^2_{adj} = 0,84 cu F(4,22) = 34,9. The predictors considered in the nonlinear regression (6.1), p, t, pt, and P, explain 86.4% of the variation in Cp.

Another nonlinear regression suggested by the Multiple Linear Regression Calculator program is the power regression given by the formula (6.2).

$$C_p = 0.121781p^{0.7014}t^{0.20496}P^{0.472451} \quad (6.2)$$

For this regression, the determination coefficient is R^2 = 0,67, and the adjusted determination coefficient has the value R^2_{adj} = 0,62, with F(3,23) = 15,41, p = 0.001. The predictors of regression (6.2) explain 66.8% of the variation of the dependent variable Cp.

LAVENDER

6.2.8. Statistical results of multivariate polynomial regressions

The multivariate polynomial regressions were obtained using the Multiple Linear Regression Calculator function from the Mathcad 15 program. In increasing order of polynomial interpolation degrees, the results shown in Table 6.2 were obtained. For the linear regression, the determination coefficient has the value R^2=0,86, and the adjusted determination coefficient, R^2_{adj} = 0,841. For the nonlinear regression (second-degree polynomial), the determination coefficient is R^2 = 0,938, and the adjusted determination coefficient R^2_{adj} = 0,905.

It was observed that for the complete second-degree polynomial, the calculated probabilities of coefficient rejection were higher than 0.05, and therefore could not be numerically represented (very large numbers). These errors show that polynomial interpolation is possible and reliable only for first-degree polynomials. One single coefficient among the 21 had a rejection probability p = 0.000251 < 0.05, namely the nonlinear term P^2, the square of the power. To obtain a linear regression, all linear terms were considered, to which the square of the power term P^2 was added. The PTC Mathcad Prime 9 program retained, in the second-degree polynomial regression, the terms included in formula (6.3). The nonlinear regression (6.3) was thus obtained.

Table 6.2

Statistical regression polynomial coefficients, rejection probabilities, and determination coefficient for the dependent variable, Cp

Term	Coeff. Gr. 1	Refusal test	Coeff.Gr. 2	Refusal test
$p^0t^0P^0$	-2.851074	0.23600	44.010454	0.070688
p	0.939278	0.01200	1.393028	0.381066
t	0.124406	0.00000	0.089719	0.298040
P	0.055325	0.00396	-0.893582	0.002561
pt	-	-	0.013858	0.167212
pP	-	-	-0.005017	0.373173
tP	-	-	0.000648	0.185367
p^2	-	-	-0.099944	0.383080
t^2	-	-	-0.000629	0.177416
P^2	-	-	0.004603	0.000851

$$C_p = 53.060209 - 0.920149P + 0.0106074pt + 0.000608567tP + 0.00460347P^2 \quad (6.3)$$

The determination coefficient of regression (6.3) is R^2=0,93, and the adjusted determination coefficient is R^2_{adj}=0,92 with F(4,22) = 74,64. The predictors considered in the nonlinear regression (6.3) explain 93.1% of the variation in Cp.

Another nonlinear regression suggested by the Multiple Linear Regression Calculator program is the power regression given by formula (6.4).

$$C_p = 0.305857p^{0,284324}t^{0,5645676}P^{0,242651} \quad (6.4)$$

for this regression, the determination coefficient is R^2=0,87 and the adjusted determination coefficient has the value R^2_{adj}=0,85, with F(3,23) = 50,11, p < 0,001. The pressure parameter, p, is not significant. The predictors of regression (6.4) explain 86.7% of the variation of the dependent variable Cp.

SAGE

6.2.13. Statistical results of multivariate polynomial regressions

The multivariate polynomial regressions were obtained using various calculation programs through the Multiple Linear Regression Calculator function of the Mathcad 15 software. In increasing order of the interpolation polynomial degrees, the results shown in table 6.3 were obtained.

It was observed that for the complete second-degree polynomial, the calculated probabilities of coefficient rejection were higher than 0.05, indicating that polynomial interpolation is possible and reliable only for first-degree polynomials. The PTC Mathcad Prime 9.0.0.0 program, following the multivariate regression analysis, retained the second-degree terms pt, tP *și* t^2. The nonlinear regression (6.5) was obtained, having the determination coefficient R^2 = 0,74 and R^2_{adj} = 0,7 (F(3,23) = 21,42).

$$C_p = 15.831455 + 0.0184703pt + 0.000796597tP - 0.000826513t^2 \quad (6.5)$$

It is observed that the multivariate regression analysis program selects the final form based on the coefficients with the lowest rejection probability, as can be seen in the complete results of the Multiple Linear Regression Calculator program shown in Table 6.3. The PTC Mathcad Prime 9.0.0.0 program provides the results for each calculation stage, and the results obtained from the Multiple Linear Regression Calculator correspond to the first calculation stage of the multivariate analysis program.

The determination coefficient for the linear regression obtained using the first-degree interpolation polynomial with the Multiple Linear Regression Calculator is 0.65, while the adjusted determination coefficient is R^2_{adj}=0.604. For the second-degree interpolation polynomial, a determination coefficient value R^2=0.776 was obtained, with an adjusted determination coefficient of R^2_{adj}=0.658.

Table 6.3

Statistical regression polynomial coefficients, rejection probabilities, and determination coefficient for the dependent variable, Cp

Term	Coeff. Pol. Gr. 1	Refusal test	Coeff. Pol. Gr. 2	Refusal test
$p^0t^0P^0$	5.394167	0.095927	5.117111	0.387644
p	1.656444	0.000273	-2.469139	0.372563
t	0.042085	0.003452	-0.005731	0.392874
P	0.066636	0.002225	0.382536	0.204218
pt	-	-	0.015967	0.209057
pP	-	-	-0.006154	0.376541
tP	-	-	0.001414	0.061869
p^2	-	-	0.275333	0.353078
t^2	-	-	-0.001052	0.118942
P^2	-	-	-0.002031	0.160615

Another nonlinear regression suggested by the PTC Mathcad Prime 9.0.0.0 program is the power regression given by formula (6.6).

$$C_p = 2.044269p^{0,382214}t^{0,145835}P^{0,260955} \quad (6.6)$$

for this regression, the determination coefficient is R^2=0,66 and the adjusted determination coefficient is R^2_{adj} = 0,61. It is obtained F(3,23) = 14,81, p < 0,001. The cutting size parameter is not significant. The predictors of regression (6.6) explain 65.9% of the variation of the dependent variable Cp.

CHAPTER 7
GENERAL CONCLUSIONS. PERSONAL CONTRIBUTIONS. NEW RESEARCH DIRECTIONS

7.1. General conclusions regarding the theoretical and experimental research

Medicinal and aromatic plants have been used for centuries for their therapeutic, nutritional, and cosmetic properties, but scientific interest today focuses on the efficient extraction of active principles they contain.

It has been found that environmental factors (temperature, light, soil moisture or carbon dioxide levels) directly influence the accumulation of active compounds in plants, which has a significant impact on the quality and quantity of the final extract.

At the same time, the analysis of extraction methods revealed a clear transition from classical techniques, such as maceration or gravitational percolation, to modern methods that allow more precise control of the process and higher efficiency. Among these, ultrasound-assisted extraction and pressure percolation have been noted for their ability to shorten processing time, reduce energy consumption, and increase the yield of valuable compound extraction.

Along with technological progress, recent studies show that mathematical modeling and experimental optimization have become essential tools in designing extraction processes. Methods such as the response surface methodology (RSM) and other types of experimental design allow for the fast and efficient determination of optimal extraction conditions, considering variables such as time, temperature, pressure, ultrasound, and the liquid-solid ratio. In some cases, these models have been extended using artificial intelligence to obtain automatic predictions and adjustments of the process, aiming to maximize yield and energy efficiency.

To achieve the objectives and conduct the experiments, the following steps were necessary:

- *Provision of appropriate measuring equipment and devices*, such as :
  - Shimadzu MOC63u thermobalance, HERBCUT TIMATIC equipment for cutting, inclined conveyor, and plant sorter;
  - TIMATIC Duo percolator and Hielscher UP400St sonicator for extraction;
  - UV-VIS spectrophotometer Jasco V-550, RETSC sieve system, precision balances KERN and Chauvin Arnoux C.A 8334 for measurements.
- *Preparation of plant material*:

- the plants were cut and sorted by size, then introduced into the extraction equipment for testing.

- *Preliminary tests – selection of the extraction method*: five extraction methods were compared:
  - pressure percolation at 5 bar;
  - pressure percolation at 7 bar;
  - pressure percolation at 5 bar assisted by ultrasound;
  - pressure percolation at 7 bar assisted by ultrasound;
  - ultrasound-assisted extraction.

The results of these tests showed that the most efficient method, for all three plants studied, was pressure percolation at 7 bar assisted by ultrasound.

- *Optimization of technological parameters*, to achieve maximum efficiency, preliminary tests were performed at different cutting sizes and material quantities, as follows:
  - Cutting size: 12 / 16 / 30 mm;
  - Plant material mass: 200 / 400 / 600 g.

The best efficiency-to-energy consumption balance was obtained at a cutting size of 30 mm and a plant material mass of 400 g

- *Evaluation of ultrasound influence*:
  - Tested ultrasound powers: 80, 100, 120, 140, 160 W;

The optimal ultrasound power was 120 W for nettle and lavender, and 100 W for sage. Since the difference between 100 W and 120 W for sage was insignificant, 120 W was selected as the standard power for all plants.

- *Complex tests with selected parameters*: at 30 mm and 400 g, the following parameters were varied:
  - Pressure: 4 / 6 / 7 bar;
  - Extraction time: 60 / 90 / 120 min;

The optimal combination for all plants was 7 bar and 120 min.

- *Extraction results*: Using the optimal combination of pressure and time (7 bar / 120 min), significant increases were achieved in the active compound content, with moderate energy consumption, as follows:
  - for *NETTLE*: +108%, maximum 13,71 mg GAE/100 g, consumption >17%;
  - for *SAGE*: +43%, maximum 30,79 mg GAE/100 g, similar consumption;
  - for *LAVENDER*: +18%, maximum 25,91 mg GAE/100 g, proportional relationship between yield and consumption.

- *Additional observations*:
    - Cyclic percolation with pressure oscillations accelerated mass transfer;
    - Cutting size influenced yield, as higher energy consumption was recorded for smaller particles;
    - Total energy consumption of the equipment increased from 0.450 kWh (5 bar, 80 W) to 0.528 kWh (7 bar, 120 W), an increase of ~17%, while extraction efficiency grew much more significantly.

7.2. Personal contributions

The present thesis includes a series of original contributions, both theoretical and experimental, carried out within the research on improving extraction equipment for active principles from medicinal plants using modern methods. The main personal contributions are as follows:

- *Development of a documentary synthesis* on the chemical composition, bioactive properties, and industrial importance of three selected medicinal plants: nettle, lavender, and sage;
- *Execution of a comparative analysis of traditional and modern extraction methods*, with a focus on pressure percolation and ultrasound-assisted extraction, emphasizing the advantages of integrating these methods into a hybrid system;
- *Establishment of preliminary working conditions* for conducting experimental tests by evaluating the influence of cutting size (12 / 16 / 30 mm) and plant material mass (200 / 400 / 600 g) to select appropriate variants for testing the technological parameters;
- *Analysis of the influence of essential technological parameters* (ultrasound power: 80 / 100 / 120 / 140 / 160 W; pressure: 5, 6, and 7 bar; extraction time: 60 / 90 / 120 min) on the extraction process efficiency, applied to all three selected plant species;
- *Establishment of the specific operating cycle for pressure percolation*, consisting of static phases (4 minutes at constant maximum pressure) and dynamic phases (6 minutes with pressure oscillations at 10-second intervals), applied repeatedly to enhance the extraction process;
- *Identification of optimal technological parameters* ensuring high extraction yield and balanced energy consumption for all three analyzed plants: pressure: 7 bar; time: 120 min; ultrasound power: 120 W. The efficiency of the hybrid method (pressure percolation assisted by ultrasound) was evaluated from both a technological and energy perspective, relative to the initial chemical composition of the plant material;

- *Highlighting of the behavior of medicinal plants* during the extraction process, showing the percentage increase in extracted active compounds (up to 108% for nettle) correlated with energy consumption;
- *Development of a mathematical model* that establishes the relationship between technological parameters and process efficiency;
- *Identification of an optimal operating regime for the extraction process* to obtain maximum efficiency in extracting bioactive compounds from medicinal plants.

The results of the research were disseminated through the publication of 6 ISI-ranked articles, 2 papers published in BDI-indexed journals (1 indexed in Scopus), and 4 papers published in the proceedings of international conferences (BDI):

1. **A-M. Tăbărașu**, N-V. Vlăduț, F. Nenciu, P. Cârdei, I. Găgeanu, L. Catană, M. Begea, M-G. Matache, D-N. Anghelache, I-C. Persu, T-A. Oncescu. Improved Hybrid Percolation–Ultrasound Extraction of Bioactive Compounds and Their Application as Nettle and Sage-Derived Biostimulants in Tomato and Pepper Crops. FOODS, 2025, 14(22), 3900; https://doi.org/10.3390/foods14223900, (FI = 5,1).
2. **Tăbărașu, A.-M**., Anghelache, D.-N., Găgeanu, I., Biriș, S.-Șt., Vlăduț, N.-V. (2023). Considerations on the Use of Active Compounds Obtained from Lavender. Sustainability, 15, 8879, https:// doi.org/10.3390/su15118879, (FI = 3,9).
3. **Tăbărașu, A.-M**., Nenciu, F., Anghelache, D.-N., Vlăduț, V.-N., Găgeanu, I. (2024). Hybrid Percolation Ultrasound Method for Extracting Bioactive Compounds from Urtica dioica and Salvia officinalis. Agriculture, 14, 1561, https://doi.org/10.3390/agriculture14091561, (FI = 3,3).
4. **Tăbărașu A-M**., Vlăduț N-V., Găgeanu I., Nenciu F., Cismaru M-E., Oncescu T-A., Gheorghe G-V., Harabagiu A., Anghelache D-N., Cujbescu D., Voicea I. (2025). Extraction and valorization of active principles from medicinal plants – a perspective for the sustainable development of farms in Romania. INMATEH Agric. Eng., vol. 77, no.3, pp. 126-146, https://inmateh.eu/archive/volumes, (FI = 0,7).
5. **Tăbărașu, A-M**., Găgeanu, I., Vlăduț, N-V., Matache, M-G., Anghelache, D-N. (2024). Experimental research on the extraction of polyphenols from nettle, lavender, and sage using the percolation method. INMATEH Agric. Eng., vol. 73, no.2, pp. 678-687, DOI: https://doi.org/10.35633/inmateh-73-58, (FI = 0,6).
6. **A.-M. Tăbărașu**, I. Găgeanu, D.-N. Anghelache, L. Catana, N-V. Vlăduț F. Nenciu. Evaluation of the biostimulatory effects of nettle and sage extracts on the development of green beans. 50th Symposium "Actual Tasks on Agricultural Engineering", Opatija, Croatia, 2025, pg. 295-304, (FI ≥ 0).

7. **A-M. Tăbărașu**, N-V., Vlăduț, P. Cârdei, D-N.Anghelache, N-A. Vanghele, M. Cristea. Statistical Modeling of the Hybrid Percolation–Ultrasound Method for Extracting Bioactive Compounds from Lavender. U.P.B. Sci. Bull., Series D, 2025, vol. 87, Iss. 4, pp. 325-340 .
8. **Tăbărașu, A-M**., Biriș, S-Șt., Vlăduț N-V., Anghelache, D-N., Zamfir, L-C., Găgeanu, I., Marin, F., Hâncu, I. (2022). Influence of environmental factors on the growth of medicinal and aromatic plants. AGRI INMA Sustain. Agric. Environ. Prot., vol.1, no. 1, p. 75-88.
9. **Tăbărașu, A-M**., Biriș. S-Șt., Găgeanu, I., Anghelache, D., Bălțatu, C., Persu, C. (2021). Methods of extracting the active principles from medicinal and aromatic plants. A review. Int. Symp. ISB INMATEH Agric. Mech. Eng., p. 88-95, ISSN 2537 – 3773.
10. **Tăbărașu, A-M**., Biriș, S-Șt., Begea, M., Anghelache, D-N., Constantin, A-M. (2022). The use of medicinal and aromatic plants in agriculture to fight pests but also as fertilizers. A review. Int. Symp. ISB INMATEH Agric. Mech. Eng., ISSN 2344 – 4118, 84-91.
11. **Tăbărașu A-M**., Anghelache D-N., Găgeanu I., Biriș S-Ș., Constantin A-M., Marin F., Hâncu I. - Mathematical models for the extraction of volatile oils and active principles from medicinal and aromatic plants. A review, TECHNICUM 2023, pg. 74-79, Vol. 14 (2023): Special Issue of the 11-th International Conference on Thermal Equipment, Renewable Energy and Rural Development (TE-RE-RD 2023), https://techniumscience.com/index.php/technium/issue/view/152.
12. **Tăbărașu, A-M.,** Vlăduț, N-V., Catană, L., Găgeanu, I., Anghelache, D-N., Nae, G., Vlăduțoiu, L., Mircea, C. (2024). Benefits of using active principles from medicinal and aromatic plants for agriculture. Int. Symp. ISB INMATEH Agric. Mech. Eng., ISSN 2344 – 4118, 24-29.

The research activity was also recognized by obtaining 5 awards at the Invention Salons, for the work entitled Innovative *Technologies for Increasing the Extraction Yield of Active Principles from Medicinal and Aromatic Plants*:

1. **Gold medal**, obtained at the *Invention Salon "Festival of Innovative and Technological Transfer*", Târgoviște, November 17–20, 2025.

2. **Special prize "Student Innovation Award"**, obtained at the *Invention Salon "Festival of Innovative and Technological Transfer*", Târgoviște, November 17–20, 2025.

3. **Gold medal**, obtained at the *INFOINVENT International Salon*, 19th edition, Chișinău, Republic of Moldova, December 3–5, 2025.

4. **Gold medal**, obtained at *innoCENTA - International Exhibition of Innovation and Technological Transfer*, Timisoara, Romania, November 6-7, 2025.

5. **Silver medal**, obtained at the *UGAL INVENT Inventions Exhibition*, Galați, October 23–24, 2025.

7.3. New research directions

Based on the results obtained in this thesis, further research can be continued in the following directions:

- Testing the hybrid method (pressure percolation assisted by ultrasound) on other medicinal plants with different phytochemical profiles.
- Investigating the influence of other technological variables (solvent temperature, solvent type, number of cycles) on extraction yield.

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