EXPERIMENTAL AND STATISTICAL ANALYSIS OF A HYBRID PERCOLATION–ULTRASOUND EXTRACTION METHOD

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This paper analyzes the efficiency of a hybrid extraction method combining pressurized percolation and ultrasonic treatment. The study analyzes the influence of key technological parameters: pressure, extraction time, and ultrasound power on process performance. Experiments were carried out using dried lavender, with three values tested for each parameter: pressure (5, 6, 7 bar), time (60, 90, 120 min), and ultrasound power (80, 100, 120 W). A total of 27 combinations were evaluated. The results identified the most effective parameter combination and highlighted the importance of mechanical process control in hybrid percolation–ultrasound extraction systems.

Keywords: statistical modelling, pressure, power ultrasound, extraction time, percolation.

1. Introduction

Bioactive compounds are natural substances with biological activity, fundamental to the functional value of medicinal and aromatic plants [1, 2, 3]. The

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production of stable, bioactive compounds-rich extracts requires the use of methods that ensure efficient mass transfer while minimizing losses caused by elevated temperatures or oxidation [4, 5].

In this regard, the improvement of extraction technologies aims not only to increase process yield, but also to enhance energy efficiency, maintain system stability, and ensure adaptability to industrial processing conditions [6]. Although conventional extraction methods are well established, they often require long processing times and provide limited control over process parameters, which may negatively affect the quality and efficiency of the extraction [7–12]. For this reason, interest has increased in non-conventional techniques based on modern physical principles, such as ultrasound-assisted extraction, microwave-assisted extraction, and pulsed electric field extraction [13–17]. These methods allow the use of mechanical and physical phenomena to improve mass transfer, reduce extraction time, and to use energy more efficiently. While both percolation and ultrasound-assisted extraction have individually proven to be efficient techniques [18, 19], studies on their combined application remain limited. The synergistic influence of these two methods on extraction efficiency and process performance is still insufficiently documented.

To better understand and improve such complex extraction processes, mathematical modelling and multivariate statistical analysis have become essential tools. Techniques such as polynomial regression, response surface methodology, and multivariate linear analysis allow for the identification of key process variables, the quantification of their effects, and the prediction of extraction outcomes. Recent studies have successfully applied these approaches to ultrasound-assisted extraction systems, demonstrating their effectiveness in improving process performance and extract quality [20-22].

This paper presents an innovative hybrid extraction method that integrates pressurized percolation with ultrasonic treatment, aiming to evaluate the mechanical efficiency and operational characteristics of the system. The innovation of the approach lies both in the combination of two extraction techniques and in the specific design of the percolation process, carried out in repetitive cycles composed of alternating static and dynamic phases. Experiments were conducted to assess the influence of key process parameters (pressure, time, and ultrasound power) on extraction efficiency. Based on experimental data and statistical modelling, the effects of these parameters on system performance were analysed. The results contribute to a enhanced understanding of hybrid percolation-ultrasound extraction processes and support their controlled implementation in technical applications.

2. Material and method

In this study, lavender (*Lavandula angustifolia*) was used as a model plant matrix on extraction process. The plant material originated from the experimental crops of INMA Bucharest, located in a temperate-continental climate zone. The plants were mechanically harvested at a height of approximately 15 cm above the ground. After harvesting, the plant material was naturally dried at room temperature, on sieves, with constant monitoring of moisture content. Drying was considered complete when the moisture level reached 9.84%, as measured with a Shimadzu MOC63u analytical moisture balance. Following this, the plants were shredded using mechanical equipment, and a particle size of 30 mm was selected for the experiments. This choice was based on the fact that, although finer shredding (16 mm and 12 mm) slightly increased the amount of extracted bioactive compounds (polyphenols), the differences compared to the 30 mm value were not significant (Table 1). Moreover, for large quantities of plant material, finer shredding may require additional technological effort without offering a significant benefit in terms of bioactive compounds yield.

Table 1

Influence of Shredding Degree on the Total Bioactive Compounds Content

t (min)	p (bar)	P (W)	Plant material (g)	Shredding degree (mm)	*Cbc* (mg GAE/100g)
60	7	100	400	30	14.93
				16	15.89
				12	16.995

t – time; p -pressure ; P- ultarsound power; Cbc – bioactive compounds content

For the extraction of bioactive compounds, an innovative hybrid system was used, combining pressurized percolation with ultrasonic treatment.

The pressurized percolation process consists of successive cycles, each including two phases: a static phase and a dynamic phase (Fig. 1). In this study, a complete pressurized percolation cycle lasted 10 minutes, comprising a 4 minute static phase and a 6 minute dynamic phase. The cycles were carried out continuously, without interruption, and the solvent used (water) was not replaced after a fixed number of cycles, but was maintained throughout the entire extraction process.

During the *static phase*, the solvent remains stationary in contact with the plant material for a defined period, promoting the diffusion of soluble compounds into the liquid. This process occurs according to the mechanism of molecular diffusion, theoretically described by Fick's law [23].

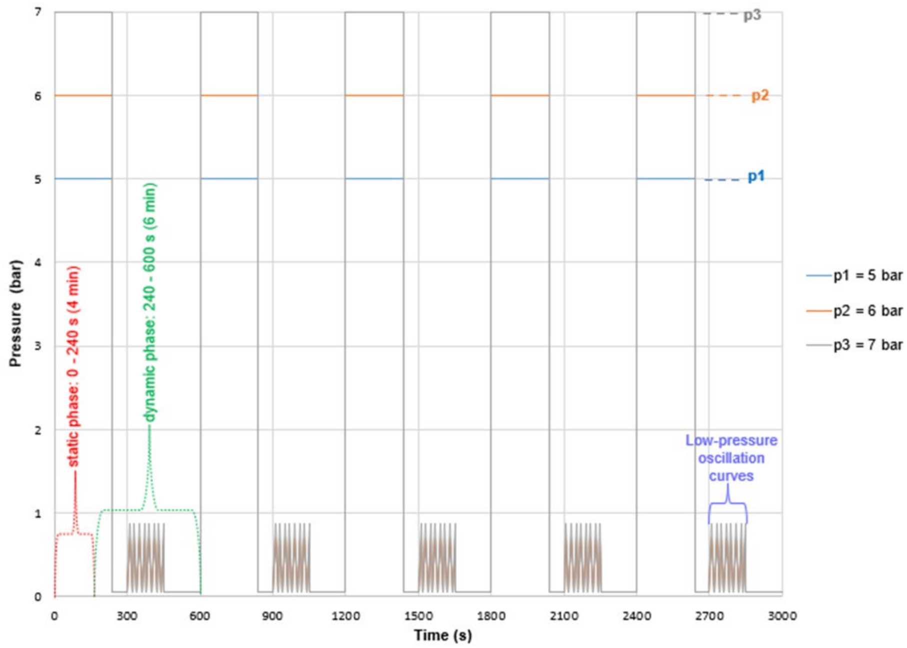

Fig. 1. Pressure diagram of the extraction process: static and dynamic phases

$$J = \frac{dm}{dtA} = -D\,\frac{\partial C}{\partial x} \tag{1}$$

where: J – diffusion flux [mol/m^2·s], dm – variation in the amount of substance [mol] over a small time interval dt [s], A – area (in this context, the contact surface between the solvent and the plant material) [m^2], D – diffusion coefficient [m^2/s], C – particle concentration [mol/m^3], x – position parameter [m].

- During the dynamic phase, the solvent is forced to circulate through the plant material by means of hydropneumatic cylinders, facilitating the dissolution and extraction of active compounds. From a theoretical perspective, the behavior of the liquid in this phase can be associated with the principles described by Darcy's law, which governs fluid flow through porous media [24]:

$$Q = -\frac{kA\Delta P}{\eta L} \tag{2}$$

where: Q – volumetric flow rate of the solvent [m^3/s], k – permeability of the porous medium (plant material) [m^2], A – cross-sectional area of the porous medium [m^2], L- length of the porous medium in the flow direction [m], ΔP – pressure difference [$\frac{\text{kg}}{(\text{m}\cdot\text{s}^2)}$], η – dynamic viscosity of the fluid [$\frac{\text{kg}}{\text{m}\cdot\text{s}}$].

The equipment used for pressurized percolation extraction is the TIMATIC Duo percolator (Tecnolab srl, Spello, Italy), which was modified from the standard configuration by removing the 24 L extraction chamber, retaining only the 12 L compartment. To provide a clearer understanding of the equipment configuration and its operating principle, a schematic representation of the TIMATIC Duo percolator is presented in figure 2. The extraction process is carried out by introducing the solvent into the extraction chamber using the double-diaphragm pump (D), while the pressure is controlled by the pneumatic valve (E). Inside the chamber, the solvent is evenly distributed through the plant material, which is compressed by the percolation piston (F), thus facilitating the solubilization of bioactive compounds. The resulting solution is collected at the bottom of the chamber, while constant pressure is maintained by the upper piston (A) and the mechanical actuator (C).

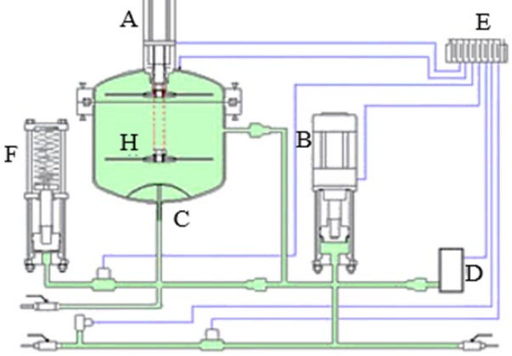

Figure 2. Schematic representation of the TIMATIC Duo percolator [25]

In this 12 L extraction chamber, the Hielscher UP400St ultrasonic probe (Hielscher Ultrasonics GmbH, Teltow, Germany) was installed, enabling the combined application of the two extraction methods on the same batch of plant material (Figure 3. a, b).

Figure 3.a. TIMATIC Duo percolator: 1 – compressor, 2 – expansion tank, 3 – extract collection vessel, 4 – control panel, 5 – extraction chamber

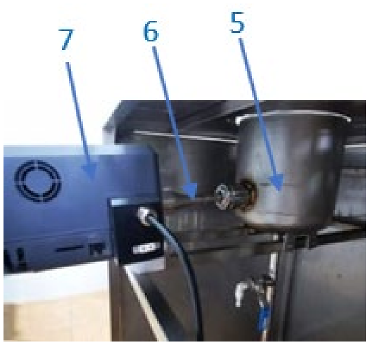

Figure 3.b. Configuration of the extraction chambre of the TIMATIC Duo percolator with the integrated Hielscher UP400St equipment: 5 – extraction chamber, 6 – sonotrode, 7 – ultrasonic generator

For each sample, 400 g of dried and shredded plant material was used, placed in permeable mesh bags, which were then positioned in the extraction chamber of the equipment (Figure 4 a, b).

Figure 4.a. Dried and shredded lavender

Figure 4.b. Placement of the permeable mesh bag with shredded lavender in the extraction chamber

For the extraction, 12 liters of water were used, introduced directly into the extraction chamber. In addition, 7 liters of buffer solvent (water) were added, which were not introduced into the extraction chamber but were used exclusively to fill the pipes and components of the system, ensuring the proper operation of the extraction equipment.

The pressures and operating times were set via the percolator control panel, while the ultrasound power was adjusted using the ultrasonic generator.

The process was carried out under varying conditions of pressure (5, 6, and 7 bar), ultrasound power (80, 100, and 120 W), and extraction time (60, 90, and 120 minutes), in order to evaluate the influence of these parameters on process efficiency.

To assess the efficiency of the extraction process, the total bioactive compounds content of the raw plant material was first determined using the Folin-Ciocalteu method, yielding a value of 28.032 mg GAE/100 g. Following the application of the hybrid extraction process, the bioactive compounds content in the extract was determined using the Folin-Ciocalteu method, resulting in 25.91 mg GAE/100 g. The extraction efficiency was approximately 92.4%, calculated as the ratio of the extracted bioactive compounds content to the total bioactive compounds content of the plant material.

3. Results and discussions

3.1. Graphical Representation of the Efficiency of Different Extraction Parameters Evaluated by the Content of Bioactive Compounds

The experimental data obtained for the total bioactive compounds content, as a function of pressure, extraction time, and ultrasound power, are presented in Table 2.

Table 2

Experimental data on the total bioactive compounds content from lavender as a function of pressure, extraction time, and ultrasound power

p (bar)	t (min)	P (W)	*Cbc* 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

To highlight the influence of technological parameters on the extraction efficiency of bioactive compounds from lavender, variations in the total bioactive compounds content were analysed as a function of pressure, extraction time, and ultrasound power. The results obtained are illustrated in Figs. 5–7.

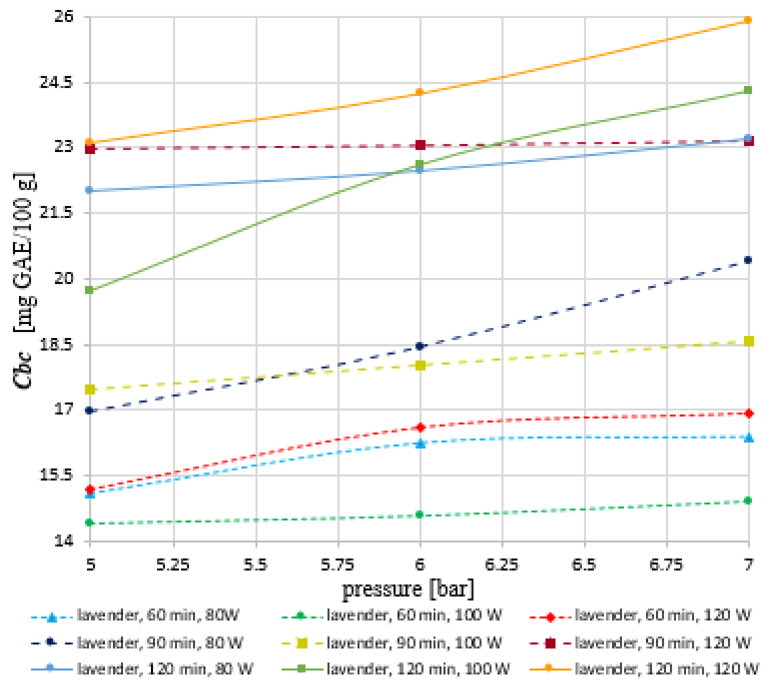

Fig. 5. Effect of pressure on extraction efficiency, assessed by bioactive compounds content (at constant time and power)

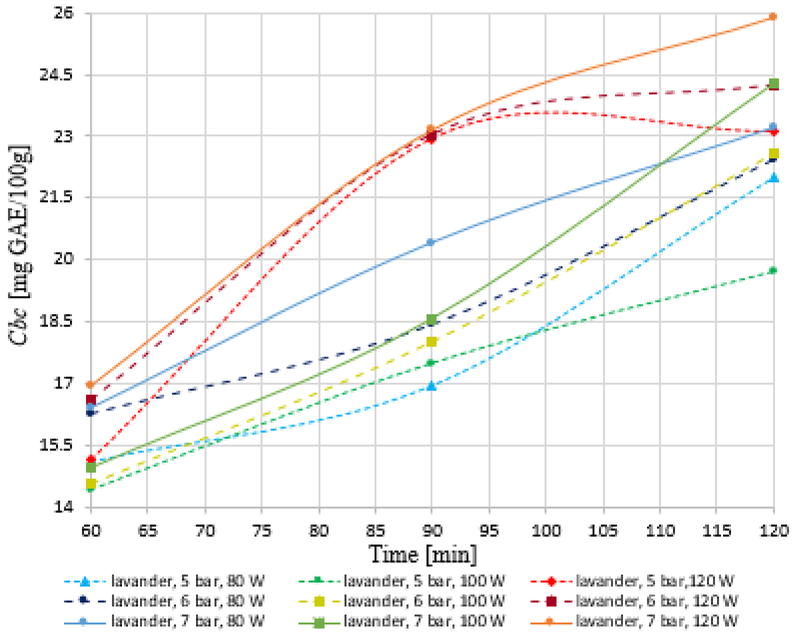

Fig. 6. Effect of extraction time on efficiency, assessed by bioactive compounds content (at constant pressure and power)

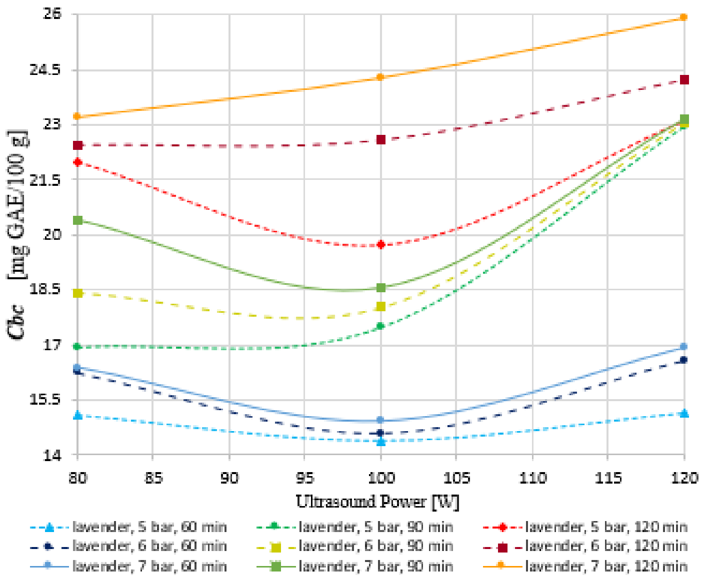

Fig. 7. Effect of ultrasound power on extraction efficiency, assessed by bioactive compounds content (at constant pressure and time)

The comparative analysis of Figures 5–7 emphasizes the cumulative impact of the technological parameters on the efficiency of the extraction process. Increases in pressure, extraction time, and ultrasonic power each contributed to improved process performance. The highest extraction efficiency of the equipment was achieved when all three parameters were simultaneously applied at elevated levels: 7 bar, 120 minutes, and 120 W, resulting in the maximum bioactive compounds content of 25.91 mg GAE/100 g. This outcome indicates a synergistic effect among the parameters and highlights the importance of improving extraction conditions to enhance the performance of the hybrid method used in this study.

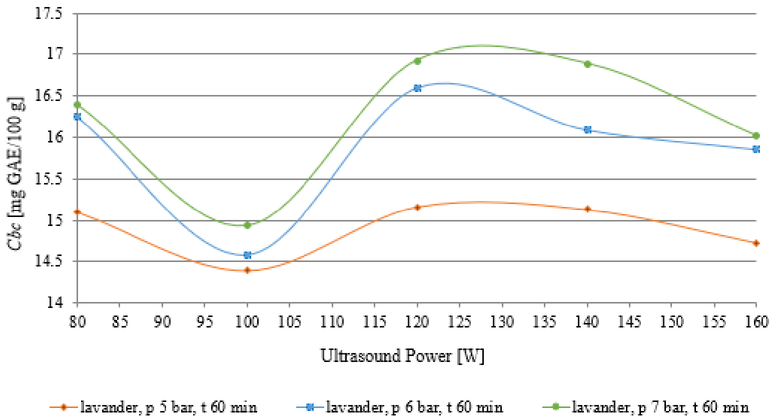

Fig. 8. Influence of *P* on *Cbc*, evaluated at five distinct ultrasound power levels

Results presented are valid for the experimental ranges investigated (5–7 bar, 60–120 minutes, 80–120 W). The pressure range was limited by the maximum capacity of the percolator (7 bar). Regarding ultrasound power, higher values (140 and 160 W) were also tested, but a decrease in extraction efficiency was observed, as illustrated in Figure 8. This may be associated with compounds degradation or cavitation instability at high intensities.

3.2. *Correlation Matrix*

Table 3

Correlations between the process variables considered in the experiments

Corelations	p	t	P	*Cbc*
p	1	0	0	0.217
t	0	1	0	0.864
P	0	0	1	0.256
Cbc	0.217	0.864	0.256	1

The independent variables (p, t, and P) are uncorrelated. The dependent variable *Cbc* shows the strongest correlation with time (t). *Cbc* is positively correlated with both ultrasound power and operating pressure.

3.3. *Results of the Linear Multivariate Analysis*

The structural dependency function for the following settings: significance level α = 0.05 (effect size = 0.39, medium effect of type f, settings according to source [26]) is given in equation (3).

$$Cbc = -2.851074 + 0.939278p + 0.124406t + 0.055325P \tag{3}$$

The results show that the set of independent variables p, t, and P is highly significant for the dependent variable *Cbc*. The coefficient of determination $R^2 = 0.86$, and the adjusted coefficient of determination $R^2_{adj} = 0.84$, indicate that these independent variables together explain 86% of the variation in *Cbc*. The analysis yielded $F(3.23) = 46.93$, $p < 0.001$. The individual predictors were further examined, and it was found that p ($t = 2.783$, $p \leq 0.011$), t ($t = 11.059$, $p < 0.001$), and P ($t = 3.279$, $p = 0.003$) were significant predictors in the model.

3.4. *Results of the Multivariate Polynomial Regression Analysis*

Using polynomial regression analysis provides more general results than those obtained from the multiple linear regression analysis presented in section 3.3. Multivariate polynomial regressions can be performed using various calculation programs. In this study, the function from source [27], implemented in Mathcad 15, was used. In ascending order of the degrees of the interpolation polynomials, the results presented in Table 4 were obtained. For linear regression, the coefficient of determination was $R^2 = 0.86$, and the adjusted coefficient of determination was $R^2_{adj} = 0.841$. For nonlinear regression (second-degree polynomial), the coefficient of determination was $R^2 = 0.938$, and the adjusted coefficient of determination was $R^2_{adj} = 0.905$.

It is observed that for the complete second-degree polynomial, the calculated probabilities for ignoring the coefficients are greater than 0.05 or cannot be represented numerically (very large values). These errors indicate that polynomial interpolation is feasible and reliable only for first-degree polynomials. Only one coefficient out of the 21 shows a rejection probability of $p = 0.000251 < 0.05$, namely the nonlinear term P^2, corresponding to the square of the ultrasound power. To obtain a nonlinear regression nonetheless, all linear terms were considered along with the square of the ultrasound power. The function from source [26] retains, in the second-degree polynomial regression, the terms presented in equation (4). The resulting nonlinear regression is given in equation (4).

Table 4

Coefficients of the regression polynomials, probabilities of being rejected, and coefficient of determination for the dependent variable *Cbc*

Regression Terms	Coeff. Deg. 1	p-value	Coeff.Deg. 2	p-value
$p^0t^0P^0$	-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

Coeff. Deg.1, Coeff. Deg.2 - polynomial regression coefficients; p-value - probability of rejection

$$Cbc = 53.060209 - 0.920149P + 0.0106074pt + 0.000608657tP + 0.00460347P^2 \quad (4)$$

The coefficient of determination of regression (4) is $R^2 = 0.93$, and the adjusted coefficient of determination is $R^2_{adj} = 0.92$, with $F(4.22) = 74.64$. The predictors considered in the nonlinear regression (4) explain 93.1% of the variation in *Cbc*.

Another nonlinear regression model suggested by [27] is the power regression presented in equation (5).

$$Cbc = 0.305857p^{0.284324}t^{0.5645676}P^{0.242651} \quad (5)$$

For this equation (5), the coefficient of determination is $R^2 = 0.87$, and the adjusted coefficient of determination is $R^2_{adj} = 0.85$. The analysis yielded $F(3.23) = 50.11$, $p < 0.001$. The pressure parameter, p, is not significant. The predictors in regression (5) explain 86.7% of the variation in the dependent variable *Cbc*.

3.5. *Minimum and Maximum Predicted Values from the Linear Statistical Model Based on Process Parameters*

The minimum and maximum values of the function *Cbc*, with three arguments, given by equation (3), were calculated using the numerical minimization and maximization functions provided in source [27]. The minimum and maximum values, for this case (multivariate linear regression of total bioactive compounds content in the extraction process from lavender plants), are presented in Table 5.

Table 5

Minimum and maximum values of total *Cbc* estimated by the multivariate linear regression (1)

Extreme values	p, bar	t, min	P, W	*Cbc*, numeric, mg GAE/100g	*Cbc*, experimental, mg GAE/100g
Minimum value	5	60	80	13.736	14.397
Maximum value	7	120	120	25.292	25.910

3.6. *Graphical Representations*

Figs. 9–14 illustrate the partial effects of one or two technological parameters on *Cbc*, using graphical representations.

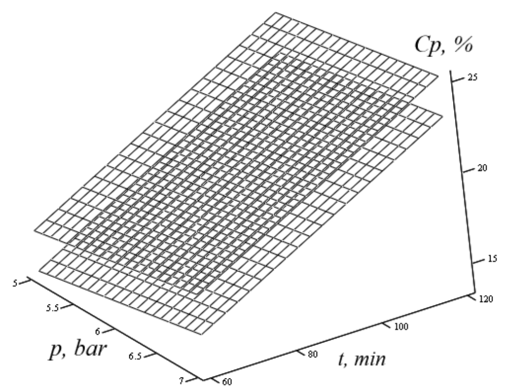

Fig. 9. Influence of pressure and extraction time on total *Cbc*, analyzed at the minimum and maximum ultrasound values of power: $Cbc(p, t, P_{min})$, $Cbc(p, t, P_{max})$

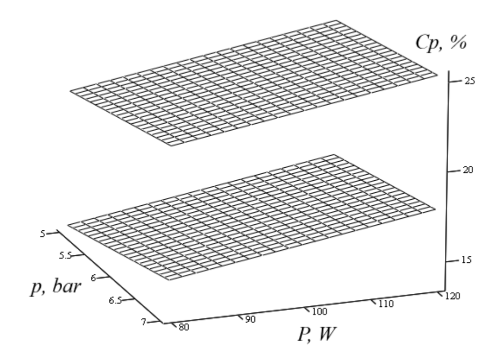

Fig. 10. Influence of pressure and ultrasonic power on *Cbc*, evaluated at the minimum and maximum extraction times: $Cbc(p, t_{\min}, P)$, $Cbc(p, t_{\max}, P)$

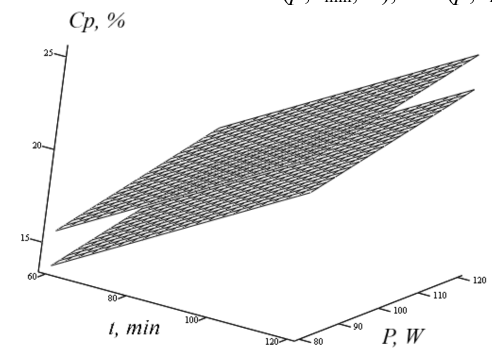

Fig. 11. Influence of extraction time and ultrasound power on *Cbc*, evaluated at the minimum and maximum values of pressure: $Cbc(p_{\min}, t, P)$, $Cbc(p_{\max}, t, P)$

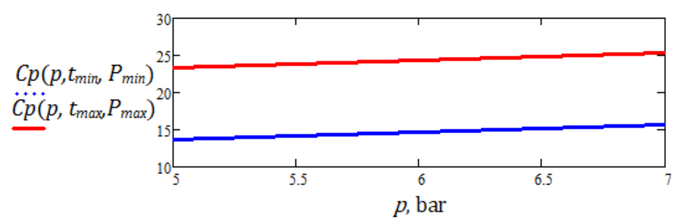

Fig. 12. Influence of operating pressure on total *Cbc*, evaluated at minimum and maximum levels of extraction time and ultrasound power, ($t_{\min}$ = 60 min, $t_{\max}$= 120 min, P_{min} = 80 W, P_{max} = 120 W)

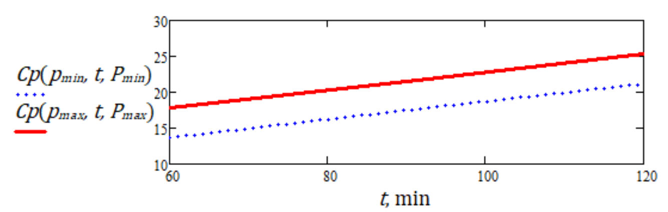

Fig. 13. Influence of extraction time on total *Cbc*, evaluated at minimum and maximum values of operating pressure and ultrasound power, (p_{min} = 5 bar, p_{max} = 7 bar, P_{min} = 80 W, P_{max} = 120 W)

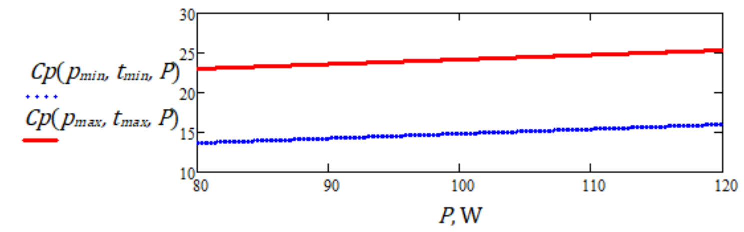

Fig. 14. Influence of ultrasound power on total *Cbc*, evaluated at minimum and maximum values of operating pressure and extraction time, (p_{min} = 5 bar, p_{max} = 7 bar, t_{min} = 60 min, t_{max}= 120 min)

Figures 9–14 emphasize the significant influence of the technological parameters: pressure, extraction time, and ultrasound power on the performance of the hybrid extraction process. The minimum and maximum process conditions, namely (p_{min}, t_{min}, P_{min}) and (p_{max}, t_{max}, P_{max}), correspond to the lowest and highest extraction efficiency observed within the tested range. These graphical representations confirm that increasing each individual parameter leads to improved extraction performance, indicating a good fit between the proposed statistical model and the experimental data, as well as a predictable trend within the tested range. No limiting effects or significant deviations were observed within the tested operating range, suggesting that the method functions in a stable and efficient manner. Further research beyond this range could provide additional useful information for refining the model and extending its technological applicability.

4. Conclusions

The results of this study show that the hybrid extraction method, which combines pressurized percolation with ultrasound treatment, is a robust and efficient approach for enhancing extraction through mechanical means. Higher values of pressure, extraction time, and ultrasound power consistently improved the process, highlighting the key role of these parameters in the system's overall performance. The highest efficiency was obtained at 7 bar, 120 minutes, and 120 W, resulting in a total bioactive compounds content of approximately 26 mg GAE/100 g. In comparison, using the lowest tested values—5 bar, 60 minutes, and 80 W—led to a content of only 15.1 mg GAE/100 g. This means that the best parameter combination increased the extraction yield by about 72% compared to the minimum settings. This result highlights the strong combined effect of pressure and ultrasound, which work together to break plant cells and help release useful compounds.

Among the statistical models applied, the second-degree polynomial regression proved to be the most suitable for describing the influence of technological parameters on the total bioactive compounds content extracted from lavender. With a coefficient of determination $R^2 = 0.931$ and an adjusted $R^2 = 0.92$, this model demonstrated superior predictive capability compared to the multivariate linear regression. The high statistical significance of the quadratic term associated with ultrasound power highlights the nonlinear behaviour of this parameter, while extraction time and pressure also made significant contributions. These results support the use of the nonlinear model as a basis for optimizing the extraction process.

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