PRELIMINARY COMPARATIVE STUDY OF THE EXTRACTION OF PHENOLIC COMPOUNDS WITH WATER FROM YERBA MATE BY NON-ISOTHERMAL SUBCRITICAL AND CONVENTIONAL ISOTHERMAL PROCESSES

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With the growing interest of the industries in phenolic compounds-rich aqueous extract, new extraction methodologies must be evaluated in terms of their technical and environmental efficiencies. In the current work, the extraction of phenolic compounds from yerba mate is evaluated using the non-isothermal subcritical water extraction technique. The extraction was conducted with a solid-liquid ratio of 0.5 g 100 mL^{-1}, with a final temperature of 120 °C and a heating ramp of 5 °C min^{-1}. Additionally, a conventional batch extraction at 70 °C was conducted in a non-pressurized system, and the energetic demand for the production of 1 m^3 of crude yerba mate extract for both scenarios was estimated. Both the extraction methods presented a similar concentration of phenolic compounds (up to 527 mg GAE L^{-1}), with a total extraction time of 161 and 128 min for the conventional and non-conventional extraction processes, respectively. The non-conventional process resulted in an increase of 111 % in the energy consumption of the conventional one, which suggests that the batch extraction conducted with water at 70 °C is more environmentally efficient under the conditions evaluated.

KEYWORDS: SOLID-LIQUID EXTRACTION, *ILEX PARAGUARIENSIS*, PRESSURIZED EXTRACTION, ENERGETIC CONSUMPTION.

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1. INTRODUCTION

The use of plants for the production of aqueous extracts has considerably grown all over the world. Such extracts are widely used in several industries, such as food, cosmetics, and pharmaceuticals, especially due to the presence of bioactive molecules such as the phenolic compounds (PC) (ARANTES et al., 2025; KIENTECA et al., 2025). Brazilian vast flora is a considerable source of PC, and several native biomasses have already been evaluated for this purpose: peach palm fruits (MONTEIRO et al., 2022), pinhão coats (MALTA et al., 2023), or yerba mate leaves (DOMINGUES et al., 2024; RODRIGUEZ et al., 2023).

Among these biomasses, yerba mate (*Ilex paraguariensis*) stands out as an efficient source of PC: the plant presents high levels of these compounds (DOMINGUES et al., 2024); it is not seasonal, which enables its availability throughout the year; and it presents a high production yield in the country (441,840 t yerba mate green leaves were produced in 2022 in Brazil) (IBGE), highlighting how accessible it is for industrial processing.

Yerba mate (YM) presents several PC in its composition (ÁVILA et al., 2025), which are beneficial to the human health for its antioxidant, antitumor, and antimicrobial activities (BOAVENTURA et al., 2015; COSTA et al., 2017; GARCIA-LAZARO et al., 2020). The extraction of these compounds has been evaluated following several different extraction methods in the last years, with special attention to the non-conventional processes such as the extraction of PC from YM in a continuous packed-bed extractor (DOMINGUES et al., 2024), in a continuous packed-bed extractor with pressurized water (RODRIGUEZ et al., 2023), or in batch systems with the assistance of ultrasound technique (LÓPEZ et al., 2023).

Subcritical water extraction is an extraction technique conducted in the temperature range of 100 – 374 °C, and it is reported as a green and environmentally friendly method (SEREMET et al., 2021). Under these conditions, there are significant changes in some physicochemical properties of water (e.g., dielectric constant), which is associated with the increase on the extraction of phenolic compounds from biomasses (BENITO-ROMÁN et al., 2020). In the current work, a non-isothermal subcritical water batch extraction of PC from YM was evaluated at the temperature of 120 °C. A conventional isothermal batch extraction at 70 °C was conducted for comparison, and the energy demand was estimated for each extraction scenario.

2. MATERIALS AND METHODS

2.1 Materials

Commercial roasted yerba mate was obtained from the Brazilian company Baldo, located in São Mateus do Sul, Paraná, Brazil. Previously to the extraction essays, the material was processed into a fine yerba mate powder by grinding and sieving (48 mesh).

Extraction essays were conducted with in-house produced deionized water. Phenolic compounds were quantified using analytical-grade reagents: Folin-Ciocalteu reagent and sodium carbonate.

2.2 Subcritical water batch extraction

The extraction of phenolic compounds with subcritical water was conducted in triplicate in a 40 mL stainless steel extraction vessel with 0.15 g of the powdered yerba mate and 30 mL of deionized water (solid-liquid ratio of 5 g yerba mate L^{-1} solvent). The extraction vessel was sealed and placed in a dry block equipment (Dry Block MA 4005, Marconi, Brazil), and the dry block was programmed with a heating rate of 5 °C min^{-1} and a final temperature of 120 °C. Once the extraction vessel reached the desired temperature, the heating of the equipment was turned off, and the extraction vessel was cooled with a natural cooling step. The extraction vessel was opened when it reached 40 °C, the mixture was filtered using a qualitative paper filter, and the extract was stored in a polypropylene Falcon tube until further analysis.

The temperature *versus* time profile was continuously monitored and recorded during the subcritical water extraction process.

2.3 Conventional water batch extraction

To provide a comparison baseline to the subcritical water batch extraction, a conventional process was conducted in triplicate in an Erlenmeyer flask. Initially, 100 mL of water was added to the flask, and it was placed in a Dubnoff water bath with controlled temperature and agitation (Novatecnica, Brazil). Once the water reached the temperature of 70 °C, 0.5 g of powdered yerba mate was added to the flask, and the mixture was submitted to an agitation of 150 rpm. Periodically, approximately 1 mL of the mixture was collected, filtered, and submitted to the quantification of the phenolic compounds, until it reached a concentration similar to the concentration obtained in the subcritical water extraction. No more than 10 mL of the mixture was retrieved from the system during the sample collection.

2.4 Phenolic compounds quantification

The quantification of the phenolic compounds was conducted according to the Folin-Ciocalteu reaction methodology (SINGLETON & ROSSI, 1965), with minor modifications (reaction conducted at 23 ± 2 °C for 2 h in the absence of light), and the absorbance of the mixture was determined in a spectrophotometer (UV-Vis 1800, Shimadzu Corp., Japan) at 760 nm. A calibration curve was built with analytical-grade gallic acid (1 – 10 mg GAE L^{-1}), and it is presented in Figure 1.

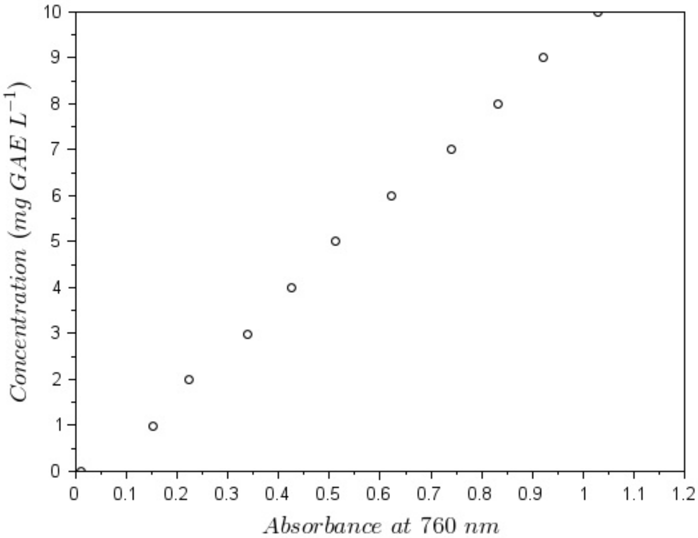

FIGURE 1. CALIBRATION CURVE USED IN THE QUANTIFICATION OF THE PHENOLIC COMPOUNDS.

The data used to build the calibration curve was fitted to a linear equation using the software Microsoft Excel, resulting in Eq. 1, and the data fitting presented a coefficient of determination (R^2) of 0.998.

$$C(mg\ GAE\ L^{-1}) = 9.993\ Abs(760\ nm) - 0.277 \quad (1)$$

2.5 Energetic consumption estimation

To compare the two extraction scenarios evaluated in this work, a simulation was conducted to determine the energetic consumption of each process for the processing of 1 m^3 of extract in a single batch extraction.

For both the extraction scenarios, the following assumptions were considered:

a) The extraction vessel presents a void fraction of 0.25 (volume);

b) The extraction vessel is assumed to be a cylinder with a height-to-diameter ratio of 1.5;

c) The volume of solids is negligible;

d) The extraction vessel is loaded with water and yerba mate at 25 °C, at the atmospheric pressure of 1 atm;

e) There is no mass loss of water during the extraction process.

At this point, to produce 1 m^3 of extract, a loading of 1 m^3 of water is considered in the beginning of the process. Additionally, to achieve the desired void fraction of 0.25, an extraction vessel of 1.33 m^3 is considered. Finally, the height (1.56 m) and diameter (1.04 m) of the extraction vessel were determined considering the desired height-to-diameter ratio and the cylindric-shape of the extraction vessel.

The amount of water used in the extraction process (997 kg) was calculated considering the density of the water at 25 °C and 1 atm, presented in Table 1.

TABLE 1. PHYSICOCHEMICAL PARAMETERS OF WATER AND AIR CONSIDERED IN THE SIMULATION

Chemical species	Water	Air	References
Physicochemical properties			
Density at 25 °C and 1 atm (kg m^{-3})	997.042	-	GREEN & SOUTHARD, 2019
	-	1.169*	HAYNES, 2014-2015
Molar weight (g mol^{-1})	18.015	28.851	SMITH et al., 2016
Tc (K)	647.1	132.2	SMITH et al., 2016
Pc (bar)	220.55	37.45	SMITH et al., 2016
Cp parameters			
a	8.712	3.355	SMITH et al., 2016
b	1.25 E-03	0.575 E-03	SMITH et al., 2016
c	-0.18 E-06	-	SMITH et al., 2016
d	-	-0.016 E+05	SMITH et al., 2016
Antoine's parameters			
A	16.3872	-	SMITH et al., 2016
B	3,885.7	-	SMITH et al., 2016
C	230.17	-	SMITH et al., 2016

Density of air at 25 °C and 1 atm (*) determined with a linear interpolation of the data presented in the book (values of density of air at 1 atm and 280 K, and 1 atm and 300 K).

2.5.1 Conventional batch extraction

Two energetic demands were considered in the conventional extraction scenario: (i) the heating of the water from room temperature (25 °C) to 70 °C; and (ii) the maintenance of the temperature in the extraction vessel.

The energy required for the heating of the water from 25 to 70 °C was calculated considering Eq. 2, where Q (J) is the energy consumption, w_w (997 kg) is the amount of water used in the extraction; MM_W (kg kmol^{-1}) is the molar weight of the water, presented in Table 1, R (8.314 J mol^{-1} K^{-1}) is the universal gas constant, $Cp(T)$ (J kmol^{-1} K^{-1}) is the heat capacity of the water, and T_o and T_f (298 and 343 K) are, respectively, the initial and final temperature.

$$Q = \frac{w_W}{MM_W} R \int_{T_o}^{T_f} \left(\frac{Cp}{R}\right) dT \tag{2}$$

The heat capacity of the water was calculated considering a function of temperature, as described in Eq. 3, where a, b, and c are specific parameters for the chemical species (Table 1).

$$\frac{Cp}{R} = a + b\,T + c\,T^2 \tag{3}$$

The energy required for the maintenance of the water temperature at 70 °C during the extraction time was determined considering Eq. 4, where U (0.55 W m^{-2} K^{-1}) is a conventional value for the overall heat transfer coefficient in industrial extraction vessels, A (m^2) is the surface area of the extraction vessel, ΔT (45 K) is the difference between the temperature of the water during the extraction process (343 K) and the room temperature (298 K), and t (150 min, 9,000 s) is the extraction time.

$$Q = U\,A\,\Delta T\,t \tag{4}$$

2.5.2 Subcritical water batch extraction

In the subcritical water batch extraction, a single energetic demand was considered: the heating of the extraction vessel from 25 to 120 °C. For so, it was considered that the extraction vessel is closed and sealed at room temperature, and no mass transfer occurs between the extraction vessel and its surroundings after it is sealed. Additionally, the extraction vessel (1.33 m^3) is composed of 1 m^3 of water and 0.33 m^3 of air at 25 °C and 1 atm.

Initially, the pressure of the system was calculated as the sum of the pressure of the individual species (water and air). The pressure of the water was calculated considering Antoine's equation (Eq. 5), where P_W (kPa) is the individual pressure of water, T (120 °C) is the temperature of the process, and A, B, and C are the specific parameters for water (Table 1).

$$\ln P_W = A - \frac{B}{T + C} \tag{5}$$

The pressure of the air was calculated considering the Equation of State (EoS) of van der Waals (Eq. 6), where P (Pa) is the individual pressure of air, T (393 K) is the temperature of the system, V (m^3 mol^{-1}) is the molar volume of the air, and a' and b' are the specific parameters for the chemical species, calculated considering Eqs. 7 and 8.

$$P = \frac{R\,T}{V - b'} - \frac{a'}{V^2} \tag{6}$$

$$a' = \frac{27}{64}\frac{R^2\,T_c^2}{P_c} \tag{7}$$

$$b' = \frac{1}{8}\frac{R\,T_c}{P_c} \tag{8}$$

Where T_c (K) and P_c (Pa), presented in Table 1, are the critical temperature and pressure for the chemical species, respectively.

Once the pressure of the system was calculated, the amount of water in the vapor phase was estimated. It was considered that the liquid phase of the water presented no changes in its volume. The a' and b' parameters for the water were calculated (Eqs. 7 and 8) considering its T_c and P_c (Table 1), and the van der Waals EoS (Eq. 6) was used to determine the molar volume V of the water in the temperature of 120 °C and the pressure of 198.5 kPa, previously determined as the individual pressure of water, considering Eq. 5. The molar quantity of water in the vapor phase was then calculated considering the determined value of V, the volume of the extraction vessel, and its void fraction.

Finally, the energy required for the subcritical water extraction (Q, J) was estimated considering the difference between the final and initial enthalpies for both the water and air (ΔH_W and ΔH_{air}, J), as described in Eq. 9.

$$Q = \Delta H_W + \Delta H_{air} \tag{9}$$

The enthalpy difference for water was calculated considering Eq. 10, where m_W (kg) is the total mass of water added to the extractor vessel, H_W^o (J kg^{-1}) is the enthalpy of liquid water at 25 °C, m_W^{vap} and m_W^{liq} (kg) are the mass of water in the vapor and liquid phases, respectively, at the extraction condition, and H_W^{vap} and H_W^{liq} (J kg^{-1}) are the enthalpies of water in the vapor and liquid phases, respectively, at the extraction condition.

$$\Delta H_W = \left(m_W^{vap}\,H_W^{vap} + m_W^{liq}\,H_W^{liq}\right) - m_W\,H_W^o \tag{10}$$

The enthalpy difference for air was calculated considering Eqs. 11 – 18, where $H_{air}(T_i, P_i)$ is the enthalpy of air in the temperature and pressure of T_i and P_i, H_{air}^{gi} is the enthalpy of air as gas ideal, and H_R is the residual enthalpy of air.

$$\Delta H_{air} = m_{air}\left(H_{air}(T_2, P_2) - H_{air}(T_1, P_1)\right) \tag{11}$$

$$H_{air}(T_i, P_i) = H_{air,o}^{gi}(T_r, P_r) + \int_{T_r}^{T_i} Cp^{gi}\,dT + H_R(T_i, P_i) \tag{12}$$

$$\Delta H_{air}|_{1\to 2} = \int_{T_1}^{T_2} Cp^{gi}\,dT + H_R(T_2, P_2) - H_R(T_1, P_1) \tag{13}$$

$$Cp^{gi} = R\,(a + b\,T + c\,T^2 + d\,T^{-2}) \tag{14}$$

$$H_R(T_i, P_i) = R\,T\,(Z - 1) \tag{15}$$

$$Z^3 - (1 + B')\,Z^2 + A'\,Z - A'B' = 0 \tag{16}$$

$$A' = \frac{a'P}{R^2\,T^2} \tag{17}$$

$$B' = \frac{b'P}{R\,T} \tag{18}$$

3. RESULTS AND DISCUSSION

3.1 Extraction of phenolic compounds from yerba mate

Initially, the subcritical water batch extraction was conducted, and the profile of temperature *versus* time (Fig. 2) was determined. The system presented an average heating rate of 5.2 °C min^{-1}, and a total heating time of 21 min, from 16 °C to 125 °C. The subsequential natural cooling step was slow, during 107 min, which resulted in a total time for the extraction process of 128 min.

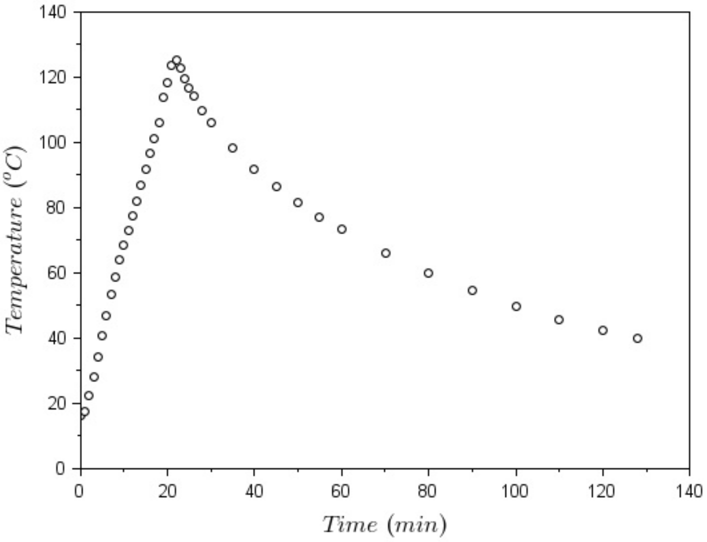

FIGURE 2. TEMPERATURE *VERSUS* TIME ON THE SUBCRITICAL WATER EXTRACTION HEATING AND COOLING STEPS.

The concentration of phenolic compounds from the extract obtained under the subcritical water batch extraction was measured and resulted in a value of 527 ± 12 mg GAE L^{-1}.

A conventional batch extraction was conducted with the same solid-liquid ratio (0.5 g 100 mL^{-1}), in a lower temperature (70 °C), which is a conventional temperature in industries of plant extracts. This temperature was selected as a control temperature, once it does not require a pressurized extraction vessel during the extraction, and the required extraction time to obtain an extract with a phenolic compounds concentration similar to the one obtained in the subcritical water extraction was determined.

The conventional batch extraction resulted in an extract with a phenolic compounds concentration of 517 ± 21 mg GAE L^{-1} after 150 min of contact of the solid with the liquid, with an additional heating time of 11 min (considering an initial temperature of 16 °C, a final temperature of 70 °C, and a heating rate of 5 °C min^{-1}), resulting in a total extraction cycle of 161 min.

The direct comparison of the two different extraction scenarios cannot be conducted, once they present significant differences in their processes. Hence, an energetic consumption estimation was conducted, and these results were used to determine if the subcritical water batch extraction presents a similar energetic efficiency to the one observed in the conventional batch extraction scenario.

3.2 Energetic consumption estimation

To determine the best extraction scenario evaluated in the current work, the energetic consumption of the processing of 1 m^3 of crude yerba mate extract in an industrial plant was estimated. Initially, the total energy demand of the conventional batch extraction scenario was calculated (Table 2).

TABLE 2. ENERGY DEMAND FOR THE CONVENTIONAL BATCH EXTRACTION SCENARIO.

Parameter	**Value**
Heating energy demand	1.88 E+08 J
Temperature maintenance energy demand	1.33 E+06 J

Among the two energy consumption steps considered (heating of the solvent from room temperature to 70 °C and maintenance of the temperature in the desired temperature during the extraction cycle), the first one is the most demanding energy process.

For the subcritical water extraction scenario, initially the pressure of the system was determined (Table 3), followed by the determination of the amount of water in the vapor phase in the vapor-liquid equilibrium (VLE) of the solvent in the extraction condition.

Table 3. Estimation of the pressure of the system in the subcritical water extraction condition.

Parameter	**Value**
Individual pressure of water (P_W)	198.5 kPa
Individual pressure of air (P_{air})	134.8 kPa
Total pressure of the system (P)	**333.3 kPa**

When considering the results presented in Table 4, it is clear that the heating of the water is considerably more energy demanding than the heating of the air. Such result is directly related to the amount of each chemical species in the extraction vessel: in the 1.33 m^3 extraction vessel, 1 m^3 of water (25 °C, 1 atm) is added to the system in the beginning of the process, which is equivalent to 997 kg of water, while the remaining volume (0.33 m^3) is the air volume, which is equivalent to a mass of 0.397 kg of air.

Table 4. Energy demand for the air and water to achieve the subcritical water extraction condition.

Parameter	**Value**
Total mass of water (m_W)	997.0 kg
Enthalpy of liquid water at 25 °C* (H_W^o)	104.5 kJ kg^{-1}
Mass of water in the vapor phase (m_W^{vap})	0.368 kg
Enthalpy of water in vapor phase at LVE** (H_W^{vap})	2,706 kJ kg^{-1}
Mass of water in the liquid phase (m_W^{liq})	996.6 kg
Enthalpy of water in liquid phase at LVE** (H_W^{liq})	503.7 kJ kg^{-1}
Enthalpy difference for water (ΔH_W)	**3.99 E+08 J**
Enthalpy difference for air (ΔH_{air})	**3.82 E+04 J**

Value of the enthalpy of liquid water at 25 °C (*) was retrieved from Aspen Plus software; and the values of enthalpy of water in vapor and liquid phases at the liquid-vapor equilibrium (**) were retrieved from SMITH et al., 2016.

Finally, the total energy demand for each scenario was calculated (Table 5). When considering the production of the same volume of crude extract (1 m^3) with a similar concentration of phenolic compounds (517 – 527 mg GAE L^{-1}), the required process time of the subcritical water extraction (128 min) is considerably lower than the conventional one's (161 min), which represents a decrease of 20.5 % of the conventional extraction scenario time. On the other hand, the energy demand of the subcritical water batch extraction is considerably higher than the energy demand for the conventional process, with an increase of 111 % of the conventional extraction scenario's energy consumption.

Table 5. Results on the conventional and subcritical water batch extraction scenarios.

Scenario	**Conventional batch extraction**	**Subcritical water batch extraction**
Extract production (m^3)	1.0	1.0
Extract concentration* (mg GAE L^{-1})	517 ± 21	527 ± 12
Temperature (°C)	70	120
Total pressure (atm)	1	3.29
Process time (min)	161	128
Energy consumption (J)	1.90 E+08	3.99 E+08
Energy cost** (R$)	33.19	69.80

Results of concentration (*) presented as average ± standard deviation. Energy cost (**) calculated considering the energy price of 0.63 R$ kWh^{-1}, retrieved from the website of the Paraná's energy distributor company Copel.

Although the results presented in Table 5 were obtained following several assumptions (e.g., a conventional value for the overall heat transfer coefficient in industrial extraction vessels, a negligible mass loss during the processes, and an extrapolation of the laboratory-scale experiment to an industrial 1 m^3 scenario), which might lead to slight differences to a real scenario, they present a preliminary evaluation that can be used to select potential engineering pathways for the production of phenolic compounds rich extracts from yerba mate. The conduction of further studies of subcritical water batch extraction at different temperatures and different solid-liquid ratio is suggested for evaluating the efficiency of the process, specially comparing the extraction process time and energy consumption with the conventional one.

4 CONCLUSIONS

The use of the conventional water extraction (70 °C, 1 atm) and the subcritical water extraction (120 °C, 3.29 atm) in the extraction of phenolic compounds from yerba mate presented a similar technical efficiency, resulting in a similar concentration for the extracts (517 and 527 mg GAE L^{-1}). The subcritical water extraction was considerably faster than the conventional one, resulting in a decrease of 20.5 % of the time of the process, which is a significant result on Engineering aspects. The energy demand of the subcritical water extraction, on the other hand, increased 111 % when compared to the conventional one, which also resulted in an increase in the energy cost (from 33.19 to 69.80 R$ for producing 1 m^3 of crude yerba mate extract). Such results were estimated considering simplifying assumptions in a scale-up simulation, and must be validated in a pilot-scale experiment.

When considering these results, the conventional extraction technique was considered the most promising one, but other subcritical water extraction conditions should be evaluated and compared to the conventional one, especially considering more unfavorable conditions, with higher solid-liquid ratios.

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