Effect of operational variables on the extraction of compounds with antioxidant capacity from chicory roots

Efecto de las variables operativas sobre la extracción de compuestos con capacidad antioxidante de raíces de achicoria

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ABSTRACT

Polyphenol solvent extraction from vegetable matrices has gained significant importance in various sectors, including food, pharmaceuticals, and agro-industries. This research focuses on the experimental design of Batch extraction procedures for obtaining polyphenols from dried and milled chicory roots. Air-forced and vacuum-drying techniques were employed to dry fresh chicory roots. The research examined the impact of operational variables on obtaining extracts from dried chicory roots. We developed a comprehensive mathematical model using diffusion transfer principles, taking into account various operational factors. We integrated the model into the optimization tool General Algebraic Modeling System (GAMS) and subsequently validated it through experimentation. The results demonstrated strong agreement between the theoretical and experimental data, with satisfactory values for both the root mean square error and correlation coefficients. The optimal extraction conditions that yielded maximum outputs were 50°C temperature, 1.10 m s^{-1} agitation speed, 50% ethanol concentration, and 20 ml solvent per gram of flour. Moreover, we observed higher diffusivity coefficients for polyphenolic compounds and lower activation energy values for extracts derived from vacuum-dried chicory root flour at 60°C and 25 mm Hg pressure. Overall, the proposed mathematical model effectively predicted the described behavior with satisfactory accuracy.

Key words: drying methods, antioxidant extraction, phenolic content, optimization model.

RESUMEN

La extracción por solvente de polifenoles a partir de matrices vegetales ha ganado gran importancia en diversos sectores, incluidos el alimentario, farmacéutico y agroindustrial. Este estudio se centra en el diseño experimental de la operación de extracción Batch para la obtención de polifenoles a partir de raíces de achicoria deshidratadas y molidas. Se emplearon las operaciones de secado al vacío y por convección forzada de aire para deshidratar raíces de achicoria fresca. Se evaluó el impacto de diversas variables operativas en la obtención de extractos a partir de raíces de achicoria deshidratadas. Se desarrolló un modelo matemático integral utilizando los principios de transferencia por difusión, considerando diversos factores operativos. Dicho modelo se implementó en la herramienta de optimización General Algebraic Modeling System (GAMS) y posteriormente se validó con resultados experimentales de laboratorio. Los resultados demostraron una fuerte concordancia entre los datos teóricos y experimentales, con valores satisfactorios tanto para el error cuadrático medio como para los coeficientes de correlación. Se encontró que las condiciones óptimas de extracción que produjeron resultados máximos fueron para la temperatura de 50°C, velocidad de agitación de 1,10 m s^{-1}, concentración de etanol del 50% y 20 ml de disolvente por gramo de harina. Además, se observaron mayores coeficientes de difusividad para los compuestos polifenólicos y valores de energía de activación más bajos para los extractos derivados de harina de raíces de achicoria deshidratadas al vacío a 60°C y 25 mm Hg de presión. En general, el modelo matemático propuesto predijo eficazmente el comportamiento descrito con una precisión satisfactoria.

Palabras clave: métodos de secado, extracción de antioxidantes, contenido fenólico, modelo de optimización.

Introduction

Polyphenol extracts derived from vegetable products have increased in importance in both the food and pharmaceutical industries due to their applications as food preservatives and their potential impact in mitigating certain diseases (Aguiñiga-Sánchez *et al*., 2020; Kaur, 2020; Mir *et al*., 2018; Pateiro *et al*., 2021; Vega-Galvez *et al*., 2023; Zhang *et al*., 2023). Studying the processing of chicory roots enables us to evaluate their use in producing flour and concentrating antioxidants that can enhance various food matrix

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antioxidant and antimicrobial capacity, improving their preservation. Whether in the form of flour or extract, the products of this process will supply applications as nutraceutical substances and prebiotics. Their consumption may help reduce the risk of cardiovascular diseases, atherosclerosis, and conditions that affect the proper functioning of the colon, thus promoting overall health (Palacios Flores, 2022). Chicory (*Cichorium intybus* L.) root is a notable consumable source of inulin, housing an array of antioxidants such as chicoric, chlorogenic, ferulic, and caffeic acids, along with tannins (Perović *et al*., 2021). Like many other vegetables and herbs, this root is prone to rapid deterioration (Sánchez-Sáenz *et al*., 2014). As a result, these materials necessitate drying procedures to facilitate processing within a preplanned annual operational timeframe, requiring equipment that permits substantial extraction as technological limitations dictate.

In this research, we tested convective and vacuum drying. The convection drying method is the most used to preserve products in the food industry. It allowed an evaluation of the behavior of the polyphenolic compounds with the drying parameters tested. The aim of vacuum drying was to study the effects on the antioxidant capacity, thus achieving shorter thermal treatment times since prolonged dehydration times can influence the quality of the final product.

In this context, the initial polyphenol composition and the quality of the flour used for extraction depend on the choice of the drying method and the associated conditions. Subsequently, the extraction process efficacy is influenced by its operational parameters and the selected drying technique (Teffane *et al*., 2021). Employing mathematical modeling serves as a robust tool to explain the intricacies of involved unit operations. Each successive stage of the process scan is intricately linked to optimize specific objectives, encompassing the minimization of resource utilization, waste generation, or expenses and the maximization of process yields while adhering to predefined quality or composition benchmarks. Such models are effectively implemented through programming techniques, culminating in nonlinear representations. Furthermore, integrating mechanistic mathematical models grounded in fundamental principles yields more realistic process simulation and optimization models.

The kinetics of solid-liquid extraction has been extensively examined in the literature concerning the extraction of antioxidants and polyphenols (Chanioti *et al*., 2014; Zhou *et al*., 2017). This exploration involves various operational factors on extraction performance, such as the solid-liquid ratio, solvent selection, particle size, and temperature, among others (Al-Farsi & Lee, 2008; Carciochi *et al*., 2018; Gubsky *et al*., 2018; Radha Krishnan *et al*., 2015; Sun *et al*., 2011). Several studies in this area have addressed the extraction process by incorporating kinetic models (Amrouche *et al*., 2019; Chaiklahan *et al*., 2014; Qu *et al*., 2010). More complex and comprehensive research has entailed the utilization of mechanistic models. An interesting example is the study by Garcia-Perez *et al*. (2010), where the authors evaluated the impact of drying temperature on antioxidant extraction by applying an optimization model. This model was devised to simultaneously ascertain the initial antioxidant concentration, as well as mass transfer and diffusion coefficients.

This study aimed to identify and optimize the conditions for extracting polyphenols from dried chicory roots using a novel mathematical model that integrates drying and extraction parameters. This approach seeks to enhance extraction efficiency and provide a versatile tool applicable to different antioxidant-rich sources, addressing a gap in existing extraction methodologies.

Materials and methods

Preparation of the samples

Fresh chicory roots (*Cichorium intybus* L.) were obtained from a local farmer from Rosario, Santa Fe, Argentina, during the autumn. The samples were subjected to drying using a convective drying oven (Tecno Dalvo, Model CHC/F/I, Argentina) and a vacuum dryer (ORL, Argentina). Each subsequently dried sample was ground using a dry blade mill (IKA, Germany) and stored under refrigeration in vacuum-sealed packaging. The resultant powdered samples were subjected to sieving to segregate particles that passed through an ASTM 40 sieve, utilizing a Ro-Tap sieve shaker (Tyler, USA). A particle diameter statistic was computed, with 0.26 mm as the mean.

The drying process was carried out in a convective oven and under vacuum. A mathematical model was used to represent the extraction process, incorporating considerations for the drying method and its associated parameters (drying temperature and air velocity or vacuum pressure) and the extraction variables (temperature, hydroalcoholic solvent composition, solid-liquid ratio, and agitation velocity). Consequently, the model established a connection between the operational parameters of both primary unit operations to attain polyphenol-enriched extracts. A series of experimental runs were conducted to determine the mass transfer coefficients. Subsequently, an independent set of experimental runs was employed to validate the proposed

model and the calculated mass transfer coefficients. Lastly, the model was optimized to maximize extraction yield. This optimization process yielded potential operating conditions suitable for scaling up the drying and extraction unit operations.

Extraction of phenolic compounds

An experimental design was carried out to study the influence of various drying and extraction variables on the yields of extractions rich in polyphenols. A fractional factorial design was chosen, considering 6 factors and 2 levels for each factor. Consequently, each design comprised 16 individual experimental runs, elucidated in Table 1.

The ranges of the selected variables for the extraction process were aligned with prior studies conducted by other researchers who had extracted polyphenolic compounds from diverse vegetable sources (Jokić *et al*., 2010; Pinelo

TABLE 1. Fractional factorial experimental design for air forced drying.

Air-forced drying						
E_{ic}	T_d (°C)	v_d (m s^{-z1})	T_e (°C)	v_e (m s^{-1})	Solvent/flour ratio, v/w (X_1)	Ethanol concentration, % (X_2)
$E1_c$	60	0.2	25	0.55	20	70
$E2_c$	80	0.2	25	0.55	30	70
$E3_c$	60	0.7	25	0.55	30	50
$E4_c$	80	0.7	25	0.55	20	50
$E5_c$	60	0.2	50	0.55	30	50
$E6_c$	80	0.2	50	0.55	20	50
$E7_c$	60	0.7	50	0.55	20	70
$E8_c$	80	0.7	50	0.55	30	70
$E9_c$	60	0.2	25	1.10	20	50
$E10_c$	80	0.2	25	1.10	30	50
$E11_c$	60	0.7	25	1.10	30	70
$E12_c$	80	0.7	25	1.10	20	70
$E13_c$	60	0.2	50	1.10	30	70
$E14_c$	80	0.2	50	1.10	20	70
$E15_c$	60	0.7	50	1.10	20	50
$E16_c$	80	0.7	50	1.10	30	50
Vacuum drying						
E_{iv}	T_d (°C)	P_d (mm Hg)	T_e (°C)	v_e (m s^{-1})	Solvent/flour ratio v/w(X_1)	Ethanol concentration, % (X_2)
$E17_v$	60	25	25	0.55	20	70
$E18_v$	80	25	25	0.55	30	70
$E19_v$	60	50	25	0.55	30	50
$E20_v$	80	50	25	0.55	20	50
$E21_v$	60	25	50	0.55	30	50
$E22_v$	80	25	50	0.55	20	50
$E23_v$	60	50	50	0.55	20	70
$E24_v$	80	50	50	0.55	30	70
$E25_v$	60	25	25	1.10	20	50
$E26_v$	80	25	25	1.10	30	50
$E27_v$	60	50	25	1.10	30	70
$E28_v$	80	50	25	1.10	20	70
$E29_v$	60	25	50	1.10	30	70
$E30_v$	80	25	50	1.10	20	70
$E31_v$	60	50	50	1.10	20	50
$E32_v$	80	50	50	1.10	30	50

Td: drying temperature; vd: air drying velocity; Pd: absolute vacuum pressure; Te: extraction temperature; ve: agitation velocity.

et al., 2005; Teffane *et al*., 2021). The drying temperature range of 60-80°C is reported by Figueira *et al*. (2004) as suitable for retaining bioactive compounds in chicory roots, further validated in a previous study by Balzarini *et al*. (2018). The levels of extraction temperature were set at 25°C and 50°C to ensure the stability of polyphenols (Bouchez *et al*., 2020; Cissé *et al*., 2012), while the ratios of solvent to flour and the concentrations of ethanol were determined based on those yielding superior extraction efficiencies (Bouchez *et al*., 2020; Carciochi *et al*., 2018; Das & Bera, 2013; Dzharov *et al*., 2016).

All extraction runs were executed employing the precise weight of dried chicory root flour dictated by the experimental design, accompanied by 600 ml of the hydroalcoholic ethanol mixture. A batch extractor within a thermostatic bath (Lauda, Alpha A6, Germany) was equipped with continuous agitation using a propeller agitator with an extraction duration of 90 min. Samples of 5 ml were taken at previously defined time intervals. They were filtered and stored in opaque containers at 3°C ± 1°C.

Total phenolic content (TPC) determination

The Folin-Ciocalteu method with modifications (Boroski *et al*., 2015) was used to determine polyphenolic compounds in samples of hydroethanolic extract of chicory roots. A 250 µl aliquot of the sample was combined with 250 µl of the Folin-Ciocalteu reagent. After 3 min, 500 µl of a saturated Na_2CO_3 solution (20%) was added. The reaction was allowed to proceed in the dark for 120 min, and the absorbance was subsequently measured at 725 nm. The results were expressed as mg of gallic acid equivalent (GAE) per 100 g of dry sample (mg 100 g^{-1} db), using a calibration curve (R^2=0.998). Total phenolic content was reported as the mean ± standard deviation, and measurements were performed in triplicate.

Initial phenolic content (TPC_0) determination

The initial polyphenol content within the chicory root flours (C_{p0}) was evaluated following the approach presented by Tao et al. (2014), which included specific changes. According to tests, a 50% hydroalcoholic mixture of ethanol was selected as the extraction solvent due to its high efficiency. To initiate the process, 10 g of chicory root flour sample was combined with 400 ml of the aqueous ethanol solution in a 600 ml beaker. The exhaustive extraction of polyphenols was conducted at 50°C with continuous agitation for 48 h. Subsequently, the resulting extract was filtered. Then, the chicory root flours were added to the beaker, and an additional 100 ml of 50% ethanolic solution was introduced. The mixture was treated at 50°C while being agitated for 4 h, followed by another round of filtration. Ultimately, the two filtrates were combined, and the TPC was ascertained utilizing the methodology elaborated in the preceding section.

DPPH radical scavenging activity determination

The 2,2-diphenyl-1-picrylhydrazyl (DPPH) method described by Shimada *et al*. (1992) was used. One ml extract was added to 5 ml of DPPH solution of 0.1 mM. The mixture was shaken for 30 s and allowed to stand for 50 min. Absorbance was measured at 517 nm using a spectrophotometer (UV-1800, Shimadzu, Japan). All samples were tested in triplicate. The results were expressed as the average value ± the standard deviation. The DPPH scavenging activity was determined using Equation 1.

$$\%\text{DPPH} = \left(1 - \frac{\text{Abs}}{\text{Abs control}}\right) \times 100 \quad (1)$$

Statistical analysis

An analysis of variance (ANOVA) was performed, and Duncan's New Multiple Range test was used ($P \leq 0.05$). The SPSS Statistical Analysis Program for Windows (SPSS Inc., Chicago, IL, USA) was used.

Mathematical model

The phenomenological description of the extraction process has undergone extensive discourse in the literature (Crossley & Aguilera, 2007; Garcia-Perez *et al*., 2010; Kostic *et al*., 2019; Popescu *et al*., 2013). In our research, the extraction model was initially employed to compute mass transfer parameters. Subsequently, they were subjected to experimental validation. Subsequently, the mathematical model was integrated with the preceding drying phase to optimize extraction yield while accounting for both drying alternatives. It is worth noting that this model does not aim to determine the superior alternative. Such a determination will be addressed in subsequent research endeavors involving assessing cost functions. Specifically, regarding extraction modeling, it is posited that the solvent permeates the solid to dissolve the extractable components, which diffuse from within the solid into the surrounding bulk liquid. This model considers the diffusion process limiting (Cheung *et al*., 2013). The model considers the following assumptions based on Fick's second law:

- Every particle was considered spherical, symmetrical, and homogeneous. So, the uniform initial distribution of the active compounds in the matrix was taken;
- The polyphenolic diffusion path was assumed only in the radial direction;

- Only the diffusion of polyphenolics was taken under study;
- The rate of diffusion of polyphenolics from the solid was independent of the time, but it depended on the temperature, explained by Arrhenius equation;
- A perfect mixing between flour and solvent was considered;
- The convective mechanism of transfer was considered from the particle surface to the solvent bulk phase;
- There are no chemical reactions or thermal degradation of the bioactive compounds in the working temperature range.

Equations

Fick's law was implemented to explain the mass transfer of polyphenols within particles using spherical coordinates in one direction and considering the assumptions:

$$\frac{(1-\varepsilon(X_1))}{D_{p,\beta}(T_e,X_2)}\frac{\partial^2 C_{p,\beta}(E_{ic},E_{iv},X_1,X_2,r,t)}{\partial t} =$$

$$(1-\varepsilon(X_1))\frac{\partial^2 C_{p,\beta}(E_{ic},E_{iv},X_1,X_2,r,t)}{\partial r^2} +$$

$$\frac{2}{r}\frac{(1-\varepsilon(X_1))\partial C_{p,\beta}(E_{ic},E_{iv},X_1,X_2,r,t)}{\partial r}, 0 < r < R_p \quad (2)$$

where (mg ml^{-1}) is the polyphenol concentration inside the particle and (m^2 s^{-1}) is the diffusivity coefficient of polyphenols within each particle. is the solvent volume fraction; is the spherical radial coordinate (m), is the time(s), and is the particle radius (m).

In this model, the diffusion coefficient of polyphenols within each particle, $D_{p,\beta}$, was obtained with the Arrhenius functionality:

$$D_{p,\beta}(T_e,X_2) = D_0(T_e) \times e^{\left(\frac{-E_A(X_2)}{R\times T_e}\right)} \quad (3)$$

where D_0 is the Arrhenius factor in m^2 s^{-1}, R represents the universal gas constant in kJ mol $^{-1}$K^{-1}, T_e is the absolute extraction temperature in K, and E_a is the activation energy of the polyphenol diffusion in kJ mol^{-1}.

The fraction of solvent volume, denoted as ε, was defined according to Equation 4, in which and were the volumes of the solid particle and the solvent phase, respectively.

$$\varepsilon(X_1) = \frac{V_\gamma}{V_\gamma + V_\beta(X_1)} \quad (4)$$

The initial condition was represented by Equation 5, assuming homogeneous initial polyphenol concentration within the particles.

$$C_{p,\beta}(E_{ic},E_{iv},X_1,X_2,r,t) =$$

$$C_{p0,\beta}(E_{ic},E_{iv}), 0 \le r \le R_p, t = 0 \quad (5)$$

The drying process impacts the concentration of polyphenols due to thermal degradation (Shad *et al.*, 2013). Consequently, distinct initial concentrations were attained based on the drying method and conditions elucidated in Table 2.

Equation 6 expressed the boundary condition for the particle center considering no mass transfer. The other necessary boundary condition was represented by Equation 7 for the interfacial polyphenols flux, where was the mass transfer coefficient in the solvent phase, was the interfacial polyphenol concentration and was the concentration in the bulk solvent.

$$\frac{\partial C_{p,\beta}(E_{ic},E_{iv},X_1,X_2,r,t)}{\partial r} = 0, r = 0, t > 0 \quad (6)$$

$$-D_{p,\beta}(T_e,X_2)\frac{\partial C_{p,\beta}(E_{ic},E_{iv},X_1,X_2,r,t)}{\partial r} =$$

$$k_{p,\gamma}(T_e,v_e,X_2)\left(C_{p,\gamma,i}(E_{ic},E_{iv},X_1,X_2,t) -\right.$$

$$\left.C_{p,\gamma}(E_{ic},E_{iv},X_1,X_2,t)\right), r = R_p, t > 0 \quad (7)$$

The equilibrium correlation between polyphenolic concentrations in both phases was depicted by Equation 8, assuming a diluted solution:

$$C_{p,\gamma i}(E_{ic},E_{iv},X_1,X_2,t) =$$

$$K(T_e,X_2)\,C_{p,\beta,i}(E_{ic},E_{iv},X_1,X_2,t), r = R_p, t > 0 \quad (8)$$

where K was the distribution constant.

The mass transfer coefficient $k_{p,\gamma}$ was computed using Equations 9 to 12, which are correlations applicable to fluidized beds of spheres within the Reynolds number range of 2–2,000 (Geankoplis, 1993).

$$S_h(T_e,X_2,v_e) =$$

$$2 + 0.95\left(R_e(T_e,X_2,v_e)\right)^{\frac{1}{2}}\left(S_c(T_e,X_2)\right)^{\frac{1}{3}} \quad (9)$$

$$k_{p,\gamma}(T_e,v_e,X_2) = \frac{S_h(T_e,X_2,v_e)\,D_{p,\gamma}(T_e,X_2)}{2\,R_p} \quad (10)$$

$$S_c(T_e,X_2) = \frac{\mu_\gamma(T_e)}{D_{p,\gamma}(T_e,X_2)\,\rho_\gamma(T_e,X_2)} \quad (11)$$

$$R_e(T_e,X_2,v_e) = \frac{2\,R_p\,\rho_\gamma(T_e,X_2)\,v_e}{\mu_\gamma(T_e)} \quad (12)$$

where S_h, S_c and R_e are Sherwood, Schmit, and Reynolds numbers, respectively.

TABLE 2. Mass transfer parameters root mean square error and R^2 values.

		$[T_e$ (°C), $X_2]$			
	Drying origin	[25, 50]	[25, 70]	[50, 50]	[50, 70]
$D_{p,\beta}$ ($m^2\ s^{-1}$)	Convection	$2.92\ 10^{-12}$	$1.37\ 10^{-12}$	$7.44\ 10^{-12}$	$3.70\ 10^{-12}$
	Vacuum	$1.03\ 10^{-11}$	$1.83\ 10^{-12}$	$1.06\ 10^{-11}$	$2.15\ 10^{-12}$
K	Convection	0.105	0.159	0.316	0.391
	Vacuum	0.248	0.166	0.596	0.416
	v_e ($m\ s^{-1}$)				
$k_{p,\gamma}$ ($m\ s^{-1}$)	0.55	$3.64\ 10^{-4}$	$3.51\ 10^{-4}$	$5.44\ 10^{-4}$	$5.24\ 10^{-4}$
	1.10	$5.12\ 10^{-4}$	$4.94\ 10^{-4}$	$7.65\ 10^{-4}$	$7.38\ 10^{-4}$
$D_{p,\gamma}$ ($m^2\ s^{-1}$)		$7.59\ 10^{-10}$	$7.28\ 10^{-10}$	$1.25\ 10^{-9}$	$1.20\ 10^{-9}$
		$[T_e$ (°C), $X_2]$			
	Drying origin	[25, 50]	[25, 70]	[50, 50]	[50, 70]
$D_{p,\beta}$ ($m^2\ s^{-1}$)	Convection	$2.92E^{-12}$	$1.37E^{-12}$	$7.44E^{-12}$	$3.70E^{-12}$
	Vacuum	$1.03E^{-11}$	$1.83E^{-12}$	$1.06E^{-11}$	$2.15E^{-12}$
K	Convection	0.105	0.159	0.316	0.391
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	v_e ($m\ s^{-1}$)				
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	1.10	$5.12\ 10^{-4}$	$4.94\ 10^{-4}$	$7.65\ 10^{-4}$	$7.38\ 10^{-4}$
$D_{p,\gamma}$ ($m^2\ s^{-1}$)		$7.59\ 10^{-10}$	$7.28\ 10^{-10}$	$1.25\ 10^{-9}$	$1.20\ 10^{-9}$

The diffusion coefficient of polyphenols in the hydroalcoholic phase, denoted as $D_{p,\gamma}$ was evaluated employing the correlation introduced by Wilke and Chang (1955), as depicted by Equation 13:

$$D_{p,\gamma}(T_e, X_2) = 1.173E10^{-16}\left(\varphi(X_2)\, MW_\gamma\right)^{0.5} \frac{T_e+273.15}{\mu_\gamma(T_e)\, V_{m,p}^{0.6}} \quad (13)$$

where ϕ, MW_γ, μ_γ, $V_{m,p}$ were solvent association parameters, molecular weight (kDa) and viscosity ($kg\ m^{-1}\ s^{-2}$) of the solvent phase, and molar volume of polyphenols ($m^3\ kmol^{-1}$).

Finally, Equation 14 resumes the mass transfer balance for the whole system:

$$(1-\varepsilon(X_1))\frac{d\langle C_{p,\beta}\rangle(E_{ic},E_{iv},X_1,X_2,t)}{dt} = -\varepsilon(X_1)\frac{dC_{p,\gamma}(E_{ic},E_{iv},X_1,X_2,t)}{dt},\ 0<t<t_f \quad (14)$$

The extraction yield, denoted as *Y*, depended on the liquid-solid ratio, the ethanol concentration used as the extracting solvent, and the initial polyphenol concentration. This efficiency is characterized by Equation 15 for the concluding extraction duration, t_e, quantifying the mass of extracted polyphenols from the mass of polyphenols initially present in the chicory root flour:

$$Y(E_{ic},E_{iv},X_1,X_2,t_e) = \frac{m_{p,\gamma}(E_{ic},E_{iv},X_1,X_2,t)}{m_{p0,\beta}(E_{id},E_{ip})}\,100 = \frac{C_{p,\gamma}(E_{ic},E_{iv},X_1,X_2,t_e)m_\gamma}{C_{p0,\beta}(E_{ic},E_{iv})\,m_\beta}\,100,\ t=t_e \quad (15)$$

The partial differential equations were discretized using the central finite difference method (CFDM) and an implicit scheme. Equations 16 and 17 defined the interval width for the radial (Δr) and temporal grids (Δt) with M = 7 and N = 10.

$$\Delta r = \frac{R_p}{G_R} \quad (16)$$

$$\Delta t = \frac{t_e}{G_t} \quad (17)$$

where G_R and G_t were the number of grid intervals in the radial coordinate and intervals in the temporal grid.

The resolution of the partial differential equations gave the local value of the polyphenol concentration. Local concentrations within the particles were integrated using

Simpson's rule to calculate average polyphenol concentrations, according to Equation 18:

$$\overline{C_{p,\beta}}(E_{ic}, E_{iv}, X_1, X_2, t) = \frac{\int_0^V C_{p,\beta}(E_{ic},E_{iv},X_1,X_2,r,t)dV}{\int_0^V dV}, t \geq 0 \quad (18)$$

The root mean square error (RMSE) minimization function was used as an objective function to solve the model. It corresponds to the sum of the differences between experimental and theoretical data.

The GAMS (General Algebraic Modeling System) program and the CONOPT tool were used to implement and solve the model. We obtained 2,283 variables and 2,269 restrictions.

Results and discussion

The development of the experimental design led to a total of 32 extraction runs. The primary subset of the dataset (consisting of 20 runs) was gathered separately to derive the mass transfer parameters. Subsequently, the remaining set of experimental runs (12 runs) was executed to validate the proposed model through the application of the estimated mass transfer parameters.

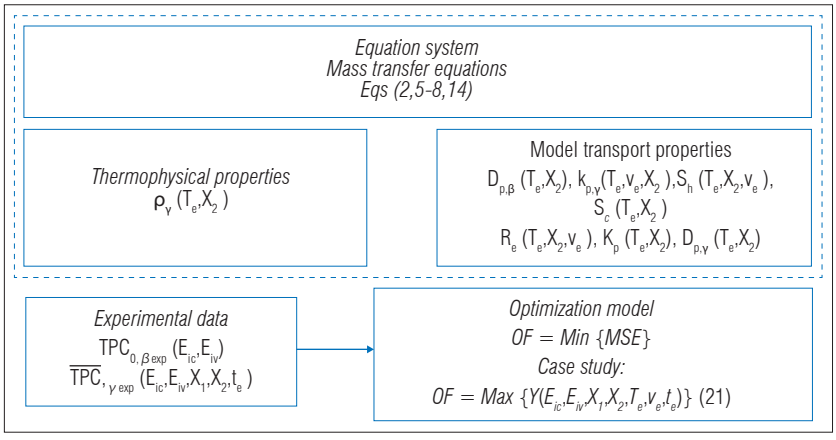
A visual representation of the mathematical model for extracting polyphenolic compounds from chicory root flour is presented in Figure 1.

Estimation of mass transfer parameters

Table 2 presents mass transfer parameter values. $D_{p,\beta}$, $k_{p,\gamma}$ and $D_{p,\gamma}$ were calculated by Equations 4, 10, and 13, respectively, and K was estimated as a model parameter.

The diverse microstructures generated by the drying treatments lead to distinct internal diffusion pathways within the particles. These differences are evident in the variations observed in the internal diffusion coefficients. Remarkably, during the extraction process, polyphenolic compounds exhibited more rapid diffusion from vacuum-dried samples than from air-forced dried samples, even when subjected to the same extraction conditions (Aravindakshan *et al.*, 2021). A lower diffusion coefficient was obtained for convective drying.

The diffusion coefficient of polyphenols, $D_{p,\beta}$, exhibits an increment with rising extraction temperatures (Chaiklahan *et al.*, 2014; Nova *et al.*, 2023; Thaisamak *et al.*, 2019; Vallejo-Castillo *et al.*, 2021). This augmentation was attributed to the molecule enhanced internal energy and increased mobility, leading to a reduction in dynamic viscosity. The activation energy values yielded by the model ranged from 20.3 to 22.4 kJ mol^{-1} for extractions from air-forced convection-dried samples and from 0.7 to 3.5 kJ mol^{-1} for vacuum-dried samples. As presented in Table 2, the highest $D_{p,\beta}$ values were observed for a bath temperature of 50°C and with an ethanol concentration of 50% in the extraction solvent ($P \leq 0.05$), regardless of the drying method. Notably, the distribution constant exhibited higher values for samples dried within the vacuum chamber, indicating a heightened extent of polyphenol extraction under these conditions. The polyphenol diffusion coefficients obtained in this study were similar to those reported by other authors for plant matrices. Chaiklahan *et al.* (2014) conducted extractions of bioactive compounds at different temperatures, with values that range from 1.07×10^{-12} m^2 s^{-1} at 50°C to 3.02×10^{-12} m^2 s^{-1} at 90°C. Aramburu *et al.*



OF: Objective function; MSE: means square error; TPC: total phenolic content.

FIGURE 1. Description of the mathematical model of extracting polyphenolic compounds from chicory root flour.

(2020) report diffusivity values of the same order (6.97×10^{-12} m² s^{-1} at 25°C).

Regarding the mass transfer coefficients of the solvent phase of Equation 10, the model provides consistent operational conditions for the extraction phase, regardless of the drying technique applied. Thus, $k_{p,y}$ is affected by agitation velocity, temperature, and the hydroalcoholic extraction mixture, while the diffusion coefficient, $D_{p,y}$ hinges on the latter two factors, as evaluated in Equation 13. These associations are detailed in Table 2.

The diffusion coefficient of total polyphenols in the phase of the solvent, the mass transfer coefficient in the solvent phase, $k_{p,y}$, and the distribution constant, K, all exhibited similar upward trends. Similarly, elevated values for the mass transfer parameters were observed at a bath temperature of 50°C and when using an extraction solvent containing 50% ethanol concentration. The mass transfer coefficient demonstrated responsiveness to agitation velocity, with higher values observed as agitation velocity increased. Consequently, higher mass transfer rates are achieved using heightened agitation velocity during extraction, elevated temperatures, and utilization of a 50% ethanol concentration within the extraction solvent. This observation aligns with published studies concerning the extraction of bioactive compounds in soybeans and certain fruits (Jokić *et al*., 2010; Velić *et al*., 2011; Zhou *et al*., 2017).

Model validation

The polyphenol concentrations from a subset of 12 experimental runs were compared with the predicted values, employing the mass transfer parameters previously determined in the model. RMSE and R^2 were calculated to assess the model predictive efficacy, obtaining values ranging between 0.002-0.013 and 0.957-0.997.

Figure 2 shows the discrepancy in the polyphenol extraction contents for samples from different drying methods. Graphs A-B and C-D correspond to samples dried in their forced convection chamber and the vacuum chamber but submitted to the same extraction conditions.

The confidence band (CB) and prediction band (PB) for total phenol contents are also presented in Figure 2. It should be noted that PB is the area where 95% of the experimental data points are expected, where all the obtained observations lie within this area. Similarly, CB is the area where 95% of the regression line was expected, which contains more than 50% of the experimental values for all experiences reported here. The obtained CB and PB fit the experimental data points well, increasing the confidence in the model's predictions. Standard probability plots of the residuals for the different experimental runs were also shown in Figure 2 (E and F). The data points above and below the line are evenly distributed, ensuring that they are typically distributed and that there are no undesirable trends or correlations between them.

The initial phenolic content of the samples dried using the air-forced convection chamber was (451.03 – 645.04) mg GAE 100 g^{-1} d.b., and for the vacuum dryer they were (805.05 – 1344.31) mg GAE 100 g^{-1} d.b. The higher polyphenol content observed with vacuum drying can be attributed to the lower operational pressures, which reduce the boiling point of water and allow for drying at lower temperatures. This minimizes the thermal degradation of polyphenols, which are sensitive to heat, thus preserving a more significant amount of these bioactive compounds. The vacuum conditions create an environment where moisture can be removed efficiently without exposing the samples to high thermal stress, aligning with observations reported in the literature (Shad *et al*., 2013; Wang *et al*., 2003).

The effects of the extraction parameters (hydroalcoholic solvent mixture, temperature, agitation velocity, and solid-liquid ratio) at the final time were examined in Figure 3. For this analysis, the drying process conditions were maintained at the lowest air temperature, air velocity, and absolute vacuum pressure values (60°C, 0.2 m s^{-1}, and 25 mm Hg).

The influence of the ethanol content on TPC is depicted in Figure 3A. The outcomes proved to be statistically significant ($P \leq 0.05$). For extracts derived from chicory roots subjected to both conventional and vacuum drying, the highest values were 428.515 ± 7.059 and 745.909 ± 4.531 mg GAE 100 g^{-1} d.b., employing a solvent concentration of 50% (v/v). Additionally, for an ethanol content of 70% (v/v), the corresponding values were 335.454 ± 2.349 and 698.518 ± 2.282 mg GAE 100 g^{-1} d.b. These findings align with the data of Thach *et al.* (2017) and Zhou *et al.* (2017).

The effect of extraction temperature on TPC was examined at 25°C and 50°C (Fig. 3B). The greatest phenolic compound concentration was achieved at 50°C, with a significant difference observed ($P \leq 0.05$). The elevation of temperature contributed to an augmented TPC, possibly attributed to the heightened diffusion coefficient leading to enhanced solubility of polyphenols in the solvent (Sarkar *et al*., 2017).

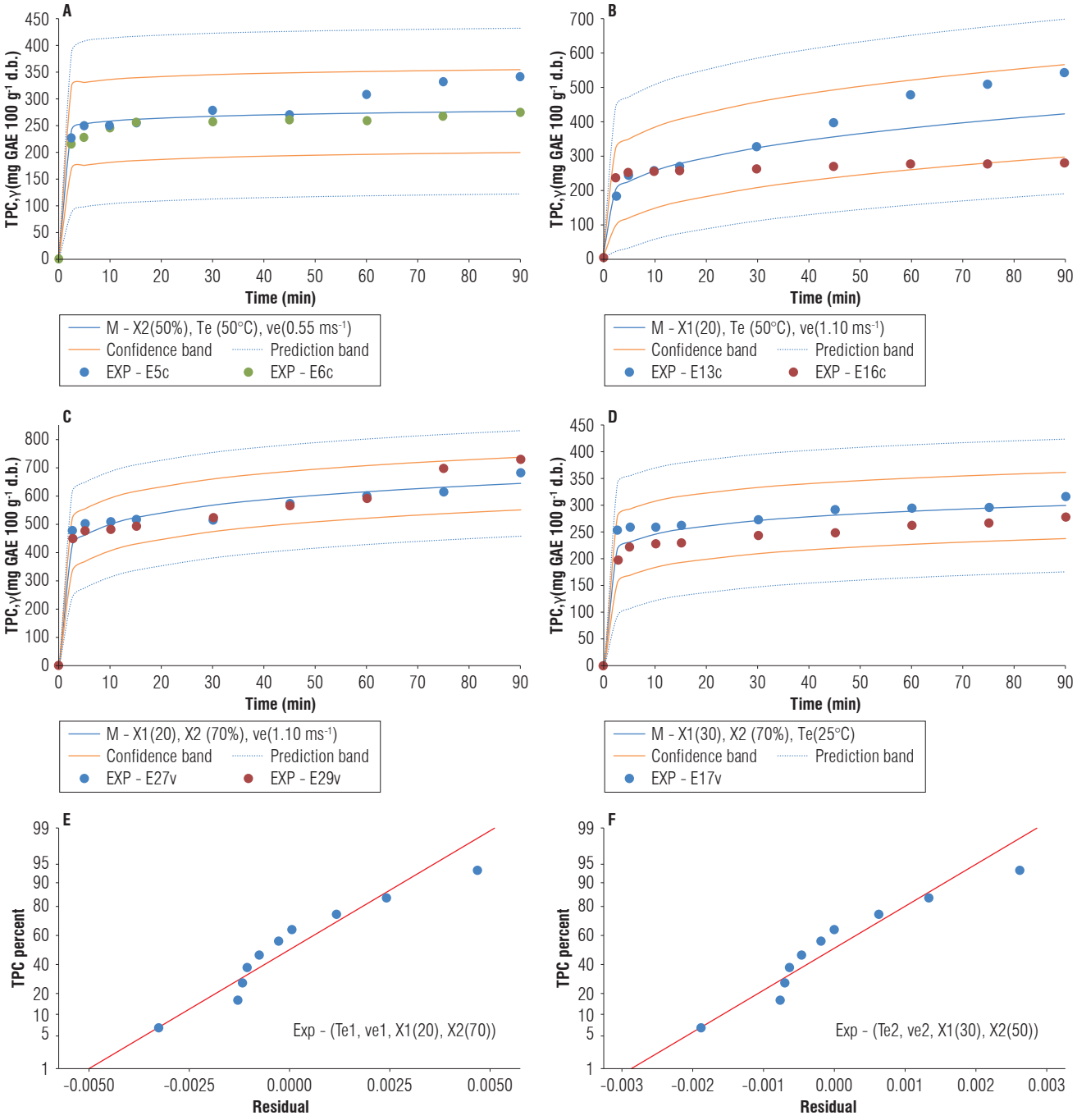


FIGURE 2. Experimental and predicted variation of the total polyphenols content (TPC) in the solvent phase for dehydrated samples using dried in the air-forced convection chamber (A and B) and vacuum chamber (C and D). Standard probability plots of residuals for different extraction operating conditions (E and F). C, convective; v, vacuum; Te, extraction temperature; ve, agitation velocity.

Figure 3C demonstrated a noteworthy effect of agitation velocity ($P \leq 0.05$) on the extracted TPC compounds within the hydroalcoholic bath. The results are consistent with those reported by Teffane *et al.* (2021). Agitation extraction techniques and velocity significantly influence total phenolic extraction (Sarkar *et al.*, 2017; Teffane *et al.*, 2021).

The effect of the liquid-to-solid ratio on the extraction of total polyphenolic compounds from ground chicory root is presented in Figure 3D, comparing ratios of 20:1 and 30:1. The highest TPC extraction was achieved with a ratio of 20:1 ($P \leq 0.05$). It is widely established that the interaction between the solvent and the solid matrix significantly

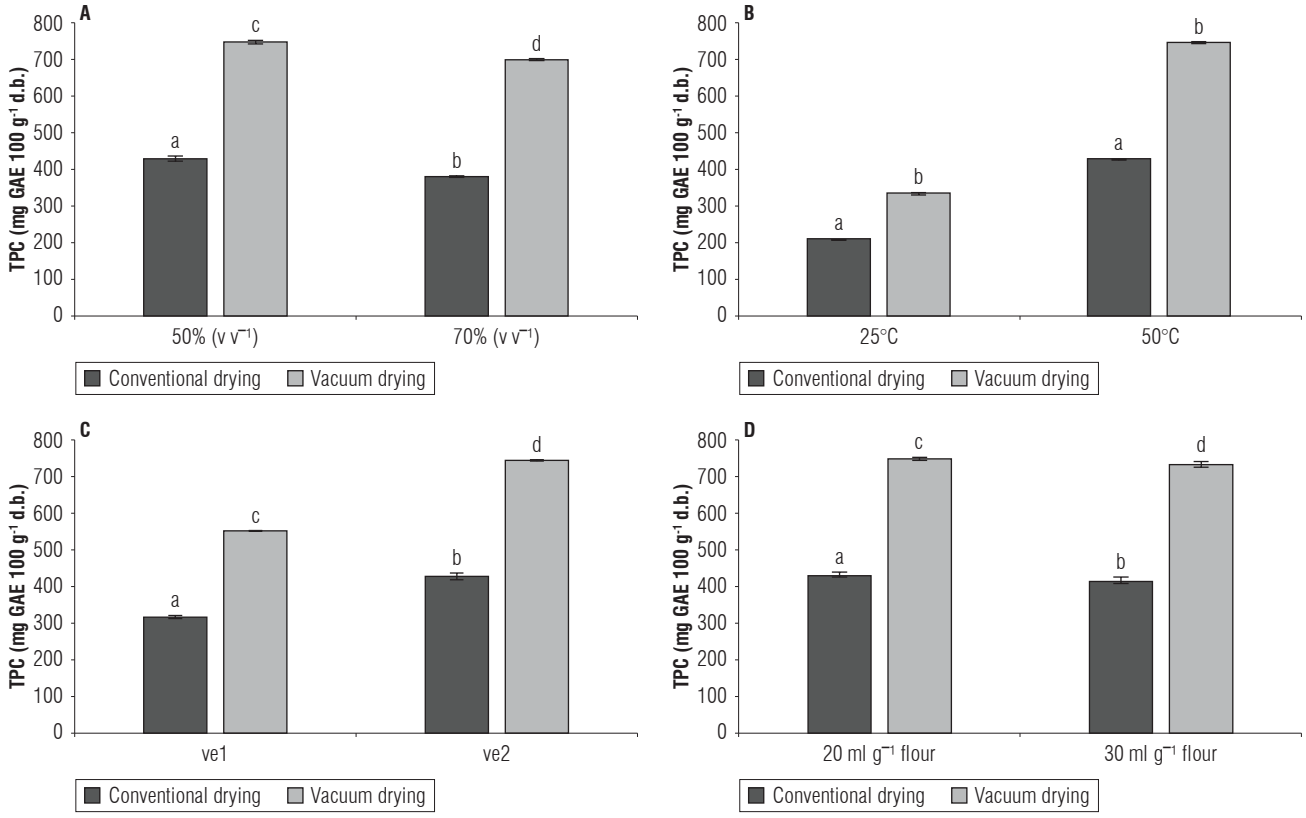


FIGURE 3. Effects of hydroalcoholic solvent mixture (A), temperature (B), agitation velocity (C), and solid-to-liquid ratio (D) on total polyphenol content (TPC) extraction from chicory roots floor. Values are expressed as mean ± SD. Different lower-case letters (a-d) represent significant differences between values according to Duncan's New Multiple Range test ($P \leq 0.05$). v v^{-1}, volume volume^{-1}; ve1, agitation velocity (0.55 m s^{-1}); ve2, agitation velocity (1.10 m s^{-1}).

influences extraction efficiency. Adequate solvent volume is essential to facilitate effective hydration and swelling of the solid phase, ultimately leading to improved recovery yield. Across all cases, parameter values were consistently higher for extracts obtained from chicory root flour subjected to vacuum drying.

The DPPH radical scavenging capacities are presented in Table 3. The maximum antioxidant activity ($P \leq 0.05$) was observed in samples subjected to vacuum drying at 60°C and 25 mm Hg absolute pressure. Elevated antioxidant activity was also evident at a dehydration temperature of 80°C. This phenomenon could be attributed to the development of Maillard reaction compounds, which enhance the antioxidant potential of chicory root flours dried at air temperatures of 80°C or above. Maillard reaction products contain electron donors and hydroxyl groups that function as reducing agents by providing hydrogen atoms to neutralize free radicals, thereby augmenting the overall antioxidant activity of the food (Vhangani & Van Wyk, 2016).

TABLE 3. DPPH of hydroalcoholic extracts of chicory roots dehydrated at 60°C and 80°C by conventional and vacuum drying. Different letters indicate that the values are significantly different according to Duncan's New Multiple Range tests ($P \leq 0.05$).

Treatment	% DPPH
60°C v1	90.876 ± 0.141[a]
60°C v2	88.591 ± 0.014[b]
80°C v1	83.552 ± 0.141[c]
80°C v2	69.327 ± 0.014[d]
60°C P1	91.035 ± 0.014[e]
60°C P2	89.497 ± 0.028[f]
80°C P1	87.151 ± 0.028[g]
80°C P2	80.104 ± 0.004[h]

DPPH (2,2-diphenyl-1-picrylhydrazyl).

Optimization model

The validated model maximized the extraction yield (% of dry weight or mg GAE g^{-1} sample). This study facilitated a comparison of operational strategies between the two

drying methods and an analysis of their impact on subsequent extraction stages.

The drying method and extraction operating conditions were considered optimization variables in the optimization model, enhancing the model degree of freedom compared to the validation model. Additionally, the initial concentrations of total polyphenols (C_{p0}) for the extraction using the drying method were integrated into the model using Equations 19 and 20. These equations represent response surfaces derived from experimental data as a function of the drying process operating conditions.

$$C_{P0,c} = 931.4 - 4.55 * T_d + 294 * V_d - 5.89 * T_d * V_d \quad (19)$$

$$C_{P0,v} = 3947.56 - 33.4317 * T_d - 22.5380 * P_d + 0.277190 * T_d * P_d \quad (20)$$

The appropriateness of the model linear response surfaces (Equations 19 and 20) was evaluated using the adjusted R^2 equations, yielding the subsequent values: Adj R^2 = 0.986 and R^2 = 0.977 for forced convection drying, and Adj R^2 = 0.999 and R^2 = 0.998 for vacuum drying. In this study, the ethanol concentration was maintained at 50%.

The optimization model was subjected to Equations 2 to 20, incorporating the objective function of maximizing the yield of total polyphenol extraction:

$$\text{OF: maximize } Y(E_{ic}, E_{iv}, X_1, X_2, T_e, v_e, t_e) \quad (21)$$

Under the objective function of Equation 21, the maximum extraction yields achieved are 63.48% and 71.19% for air-forced and vacuum drying, respectively. These optimal yields are attained by applying the same extraction operating conditions regardless of the drying method. These conditions encompass the highest extraction temperature (50°C), agitation velocity (1.1 m s^{-1}), solvent/flour ratio (30 ml g^{-1}), and maximum extraction time (90 min).

The optimal solution selected dried samples with a higher initial polyphenol concentration, obtained by employing lower air temperature, absolute vacuum pressure, or air velocity during the drying process (60°C and 25 mm Hg or 0.2 m s^{-1}). As a result, achieving maximum yields requires heightened requirements in the extraction stage, involving a higher agitation speed (1.1 m s^{-1}) and the maximum solvent-to-flour ratio (30 ml g^{-1}). The optimal extraction temperature was also consistently resolved at the upper limit (50°C), enhancing the mass transfer rate. This outcome stems from the extraction efficiency being significantly improved with increased agitation velocity and temperature, both of which approached their upper bounds due to technological constraints.

The optimization results underscored the significant impact of several processing variables on polyphenol extraction yields from both the drying and extraction stages. Further analyses involving operational and investment costs are essential to establish a balanced solution that strikes a compromise between quality and costs.

Conclusions

A phenomenological model for extracting polyphenols from chicory root flour was developed and validated, encompassing numerous extraction variables. Moreover, the model accounted for the operational variables employed in the preceding drying stage, which determined the initial concentration of polyphenols for the extraction process.

Both drying methods were included in the results to facilitate the incorporation of cost models in subsequent optimization endeavors. Consequently, this lays the foundation for an advanced optimization model that can effectively determine the appropriate drying method based on cost considerations.

The provided optimal solution captures the interdependencies among the primary critical variables. However, the model will be enhanced in future research by integrating economic and technological constraints, encompassing costs, maintenance, and operational feasibility, to select the optimal operational strategies.

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Conflicts of interest statement

The authors declare that there is no conflict of interests regarding the publication of this article.

Author's contributions

MFB conducted the research and formal analysis and prepared the initial draft. MFB and MAR developed the methodology, provided the study materials, reviewed and edited the manuscript, and supervised the planning and execution of the research activity. MAR and MCC managed

and coordinated the planning and execution of the research activity and acquired financial support for the project. All authors approved the final version of the manuscript.

Literature cited

Aguiñiga-Sánchez, I., Soto-Hernández, M., Cadena-Iñiguez, J., Suwalsky, M., Colina, J. R., Castillo, I., Rosado-Pérez, J., Mendoza-Núñez, V. M., & Santiago-Osorio, E. (2020). Phytochemical analysis and antioxidant and anti-inflammatory capacity of the extracts of fruits of the *Sechium* hybrid. *Molecules*, *25*(20), Article 4637. https://doi.org/10.3390/molecules25204637

Al-Farsi, M. A., & Lee, C. Y. (2008). Optimization of phenolics and dietary fibre extraction from date seeds. *Food Chemistry*, *108*(3), 977–985. https://doi.org/10.1016/j.foodchem.2007.12.009

Amrouche, S., Faroudja, M., & Derriche, R. (2019). Extraction of phenolic compounds from Algerian *Inula viscosa* (L.) Aiton leaves: Kinetic study and modeling. *Separation Science and Technology*, *55*(17), 3161–3174. https://doi.org/10.1080/01496395.2019.1675700

Aramburu, A., Basanta, M. F., De'nobili, M. D., & Rojas, A. M. (2020). *Aprovechamiento integral de residuos agroindustriales de cereza (Prunus avium) con alto contenido de antioxidantes naturales*. III Congreso Iberoamericano de Ingeniería de los Alimentos. Asociación de Ingenieros Alimentarios del Uruguay.

Aravindakshan, S., Nguyen, T. H. A., Kyomugasho, C., Buvé, C., Dewettinck, K., Van Loey, A., & Hendrickx, M. E. (2021). The impact of drying and rehydration on the structural properties and quality attributes of pre-cooked dried beans. *Foods*, *10*(7), Article 1665. https://doi.org/10.3390/foods10071665

Balzarini, M. F., Reinheimer, M. A., Ciappini, M. C., & Scenna, N. J. (2018). Comparative study of hot air and vacuum drying on the drying kinetics and physicochemical properties of chicory roots. *Journal of Food Science & Technology, 55*, 4067–4078. https://doi.org/10.1007/s13197-018-3333-5

Boroski, M., Visentainer, J. V., Cottica, S. M., & Morais, D. R. (2015). *Antioxidantes, principios e métodos analíticos* (1st ed.). Appris, Curitiba.

Bouchez, A., Vauchel, P., Galvan D'Alessandro, L., & Dimitrov, K. (2020). Multi-objective optimization tool for ultrasound-assisted extraction including environmental impacts. *Chemical Engineering Research and Design*, *164*, 324–337. https://doi.org/10.1016/j.cherd.2020.10.001

Carciochi, R. A., Sologubik, C. A., Fernández, M. B., Manrique, G. D., & Galvan D'Alessandro, L. (2018). Extraction of antioxidant phenolic compounds from brewer's spent grain: Optimization and kinetics modeling. *Antioxidants*, *7*(4), Article 45. https://doi.org/10.3390/antiox7040045

Chaiklahan, R., Chirasuwan, N., Triratana, P., Tia, S., & Bunnag, B. (2014). Effect of extraction temperature on the diffusion coefficient of polysaccharides from *Spirulina* and the optimal separation method. *Biotechnology and Bioprocess Engineering*, *19*(2), 369–377. https://doi.org/10.1007/s12257-013-0733-2

Chanioti, S., Liadakis, G., & Tzia, C. (2023). Solid–liquid extraction. In T. Varzakas, & C. Tzia (Eds.), *Food engineering handbook. Food processing engineering* (pp. 256–283). Routledge Handbooks Online, CRC Press, Boca Raton. https://doi.org/10.1201/b17803

Cheung, Y., Siu, K., & Wu, J. (2013). Kinetic models for ultrasound-assisted extraction of water-soluble components and polysaccharides from medicinal fungi. *Food and Bioprocess Technology*, *6*(10), 2656–2665. https://doi.org/10.1007/s11947-012-0929-z

Cissé, M., Bohuon, P., Sambe, F., Kane, C., Sakho, M., & Dornier, M. (2012). Aqueous extraction of anthocyanins from *Hibiscus sabdariffa*: Experimental kinetics and modeling. *Journal of Food Engineering*, *109*(1), 16–21. https://doi.org/10.1016/j.jfoodeng.2011.10.012

Crossley, J. I., & Aguilera, J. M. (2007). Modeling the effect of microstructure on food extraction. *Journal of Food Process Engineering*, *24*(3), 161–177. https://doi.org/10.1111/j.1745-4530.2001.tb00538.x

Das, S., & Bera D. (2013). Mathematical model study on solvent extraction of carotene from carrot. *International Journal of Research in Engineering and Technology*, *2*(9), 343–349. https://doi.org/10.15623/ijret.2013.0209052

Dzharov, V. V., Mishra, A. P., Shariati, M. A., Atanassova, M. S., & Plygun, S. (2016). Phytochemical contents in solid-liquid extraction of aqueous alcoholic extract of chicory (*Cichorium intybus* L.) leaves. *Foods and Raw Materials*, *4*(2), 32–37. https://doi.org/10.21179/2308-4057-2016-2-32-37

Figueira, G. M., Park, K. J., Brod, F. P. R., & Honorio, S. L. (2004). Evaluation of desorption isotherms, drying rates and inulin concentration of chicory roots (*Cichorium intybus* L.) with and without enzymatic inactivation. *Journal of Food Engineering, 63*, 273–280. https://doi.org/10.1016/j.jfoodeng.2003.06.001

Garcia-Perez, J. V., García-Alvarado, M. A., Carcel, J. A., & Mulet, A. (2010). Extraction kinetics modeling of antioxidants from grape stalk (*Vitis vinifera* var. Bobal): Influence of drying conditions. *Journal of Food Engineering*, *101*(1), 49–58. https://doi.org/10.1016/j.jfoodeng.2010.06.008

Geankoplis, C. J. (1998). *Transport processes and unit operations* (3rd ed.). Compañia Editorial Continental, México.

Gubsky, S., Labazov, M., Samokhvalova, O., Grevtseva, N., & Gorodyska, O. (2018). Optimization of extraction parameters of phenolic antioxidants from defatted grape seeds flour by response surface methodology. *Ukrainian Journal of Food Science*, *7*(4), 627–639. https://doi.org/10.24263/2304-974X-2018-7-4-8

Jokić, S., Velić, D., Bilić, M., Bucić-Kojić, A., Planinić, M., & Tomas, S. (2010). Modelling of the process of solid-liquid extraction of total polyphenols from soybeans. *Czech Journal of Food Sciences*, *28*, 206–212. https://doi.org/10.17221/200/2009-CJFS

Kaur, H. (2020). Constituent phytochemicals from *Catharanthus roseus*. *African Journal of Biomedical Research*, *23*(1), 9–15. https://www.ajol.info/index.php/ajbr/article/view/202130

Kostic, E., Nikolic, G. M., Mitic, S., Dimitrijevic, D., & Mitić, M. (2019). Optimization and kinetics of the solid-liquid extraction process of polyphenols from black mulberry fruit. *Revista de Chimie*, *70*(3), 853–858. https://doi.org/10.37358/RC.19.3.7019

Lutz, M., Hernández, J., & Henríquez, C. (2015). Contenido fenólico y capacidad antioxidante en frutas y hortalizas frescas y secas cultivadas en Chile. *CyTA - Journal of Food*, *13*(4), 541–547.

Mir, M. A., Kumar, A., & Goel, A. (2018). Phytochemical analysis and antioxidant properties of the various extracts of *Catharanthus*

roseus. *Journal of Chemical and Pharmaceutical Research*, *10*(10), 22–31.

Nova, C., Giraldo, L., & Cáceres-Roa, S. (2023). Extracción de polifenoles: una comparación a partir de cáscara de cacao húmeda vs cáscara de cacao secada. *Ingeniería y Competitividad, 25*(2), Article e-20612223. https://doi.org/10.25100/iyc.v25i2.12223

Palacios Flores, C. A., (2022). La achicoria en la tradición «¡A nadar, peces!»: aplicación, impacto y relevancia médica actual a través del derivado químico inulina. *El Palma de la Juventud, 4*, 97–111. https://doi.org/10.31381/epdlj.v4i4.4893

Pateiro, M., Gómez-Salazar, J. A., Jaime-Patlán, M., Sosa-Morales, M. E., & Lorenzo, J. M. (2021). Plant extracts obtained with green solvents as natural antioxidants in fresh meat products. *Antioxidants*, *10*(2), Article 181. https://doi.org/10.3390/antiox10020181

Perović, E., Tumbas Šaponjac, V., Kojić, J., Krulj, J., Moreno, D. A., García-Viguera, C., Bodroža-Solarov, M., & Ilić, N. (2021). Chicory (*Cichorium intybus* L.) as a food ingredient – Nutritional composition, bioactivity, safety, and health claims: A review. *Food Chemistry*, Article 336. https://doi.org/10.1016/j.foodchem.2020.127676

Pinelo, M., Rubilar, M., Jerez, M., Sineiro, J., & Núñez, M. J. (2005). Effect of solvent, temperature, and solvent-to-solid ratio on the total phenolic content and antiradical activity of extracts from different components of grape pomace. *Journal of Agricultural and Food Chemistry*, *53*(6), 2111–2117. https://doi.org/10.1021/jf0488110

Popescu, M., Danciu, T., Danciu, E., & Ivopol, G. (2013). Seabuckthorn oil extraction, a model for solid-liquid extraction process. *UPB Scientific Bulletin, Series B: Chemistry and Materials Science*, *75*(4), 35–42.

Qu, W., Pan, Z., & Ma, H. (2010). Extraction modeling and activities of antioxidants from pomegranate marc. *Journal of Food Engineering*, *99*(1), 16–23. https://doi.org/10.1016/j.jfoodeng.2010.01.020

Radha Krishnan, K., Azhagu Saravana Babu, P., Babuskin S., Sivarajan, M., & Sukumar, M. (2015). Modeling the kinetics of antioxidant extraction from *Origanum vulgare* and *Brassica nigra*. *Chemical Engineering Communications*, *202*(12), 1577–1585. https://doi.org/10.1080/00986445.2014.957757

Sánchez-Sáenz, C. M., Oliveira, R. A., & Park, K. J. (2014). Evaluation of chicory roots submitted to HTST drying process and its optimization. *Journal of Food Process Engineering*, *38*(1), 56–66. https://doi.org/10.1111/jfpe.12126

Sarkar, A., & Ghosh, U. (2017). Effect of extraction temperature and technique on phenolic compounds and antioxidant activity of *Tamarindus indica* seeds. *Research Journal of Recent Sciences*, *6*(2), 10–15.

Shad, M. A., Nawaz, H., Rehman, T., & Ikram, N. (2013). Determination of some biochemicals, phytochemicals and antioxidant properties of different parts of *Cichorium intybus* L.: A comparative study. *Journal of Animal and Plant Sciences*, *23*(4), 1060–1066.

Shimada, K., Fujikawa, K., Yahara, K., & Nakamura, T. (1992). Antioxidant properties of xhantan on the antioxidation of soybean oil in cyclodextrin emulsion. *Journal of Agricultural and Food Chemistry*, *40*(6), 945–948. https://doi.org/10.1021/jf00018a005

Sun, Y., Su, W., Zhang, W., Hu, Q., & Zeng, X. (2011). Optimizing the extraction of phenolic antioxidants from kudingcha made from *Ilex kudingcha* C. J. Tseng by using response surface methodology. *Separation and Purification Technology*, *78*(3), 311–320. https://doi.org/10.1016/j.seppur.2011.01.038

Tao, Y., Zhang, Z., & Sun, D. W. (2014). Experimental and modeling studies of ultrasound-assisted release of phenolics from oak chips into model wine. *Ultrasonics Sonochemistry*, *21*(5), 1839–1848. https://doi.org/10.1016/j.ultsonch.2014.03.016

Teffane, M., Boudries, H., Bey, M. B., Kadi, A., & Boukhalfa, F. (2021). Effect of solvent type, extraction temperature, agitation speed and microwave power on phenolic compound extraction and antioxidant activity of apricot kernels (*Prunus armeniaca* L.). *Current Bioactive Compounds*, *18*(1), Article e260821191386. https://doi.org/10.2174/1573407217666210215085507

Thach, N. A., & Thuy, N. M. (2017). Effect of extraction conditions on polyphenols, flavonoids, s-allyl cysteine content and antioxidant activity of black garlic extracts. *Vietnam Journal of Science and Technology*, *55*(5A), 18–25.

Thaisamak, P., Jaturonglumlert, S., Varith, J., Taip, F. S., & Nitatwichit, C. (2019). Kinetic model of ultrasonic-assisted extraction with controlled temperature of c phycocyanin from *S. platensis*. *International Journal of GEOMATE, 16*(55), 176–183. https://doi.org/10.21660/2019.55.87291

Vallejo-Castillo, V., Córdoba, E., & Panchalo, A. (2021). Modelamiento matemático de la extracción asistida con ultrasonido de compuestos bioactivos presentes en la cebolla (*Allium cepa* L.): revisión y análisis teórico. *Investigación e Innovación en Ingenierías, 9*, 112–129. https://doi.org/10.17081/invinno.9.2.4772

Vega-Galvez, A., Gomez-Perez, L. S., Zepeda, F., Vidal, R. L., Grunenwald, F., Mejías, N., Pasten, A., Araya, M., & Ah-Hen, K. S. (2023). Assessment of bio-compounds content, antioxidant activity, and neuroprotective effect of red cabbage (*Brassica oleracea* var. *Capitata rubra*) processed by convective drying at different temperatures. *Antioxidants*, *12*(9), Article 1789. https://doi.org/10.3390/antiox12091789

Vhangani, L. N., & Van Wyk, J. (2016). Antioxidant activity of Maillard reaction products (MRPs) in a lipid-rich model system. *Food Chemistry*, *208*, 301–308. https://doi.org/10.1016/j.foodchem.2016.03.100

Wang, M., Simon, J. E., Aviles, J. F., Zheng, Q. Y., & Tadmor, Y. (2003). Analysis of antioxidative phenolic compounds in artichoke. *Journal of Agricultural & Food Chemistry, 51*, 601–608. https://doi.org/10.1021/jf020792b

Wilke, C. R., & Chang, P. (1955). Correlation of diffusion coefficients in dilute solutions. *AIChE Journal*, *1*(2), 264–270. https://doi.org/10.1002/aic.690010222

Zhang, J., Netzel, M. E., Pengelly, A., Sivakumar, D., & Sultanbawa, Y. (2023). A review of phytochemicals and bioactive properties in the Proteaceae family: A promising source of functional food. *Antioxidants*, *12*(11), Article 1952. https://doi.org/10.3390/antiox12111952

Zhou, M., Chen, Q., Bi, J., Wang, Y., & Wu, X. (2017). Degradation kinetics of cyanidin 3-O-glucoside and cyanidin 3-O-rutinoside during hot air and vacuum drying in mulberry (*Morus alba* L.) fruit: A comparative study based on solid food system. *Food Chemistry*, *229*, 574–579. https://doi.org/10.1016/j.foodchem.2017.02.131