



Research Article

Obtaining *Curcuma longa* essential oil by different extraction techniques and process modeling

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Abstract

This study aims to compare the supercritical extraction (SFE) and steam distillation (SD) to obtain the *Curcuma longa* essential oil, evaluating the yield and composition for different conditions, as well as the mathematical modeling of the extraction curve. Analysis of the chemical composition of the essential oil was performed by gas chromatography coupled to mass spectrometry (GC-MS). The experimental extraction curve was plotted, and a mathematical model was used to fit the data for the SD and SFE methods. The data indicate that the best condition for SFE is 90 bar, 40 °C (0.0219 g_{oil}/g_{plant}) with 10.80% ar-turmerone and 24.12% α -turmerone, for SD the one performed at 3 bar (133 °C) (0.0486 g_{oil}/g_{plant}) with 35.42% α -zingiberene and 24.52% α -turmerone. Different major compounds were obtained varying the extraction technique. The mathematical modeling adequately represented the extraction processes.

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Steam distillation, supercritical extraction, chromatography, modeling, composition, *Curcuma longa*.

1. Introduction

Curcuma longa L is a plant of the Zingiberaceae family, whose rhizome can be used to obtain its essential oil, which is composed of mainly ar-turmerone, α and β -zingiberene [1-2]. Its essential oil has antifungal properties, as well as the ability to inhibit mycotoxins from certain fungi, making it possible to use it as a food preservative [3-5]. Another relevant property is its antioxidant capacity, which can be applied to reduce the rancidity of foods [6]. The literature also indicates the promising anticancer capacity of ar-turmerone compound [7].

To obtain the volatile extract, techniques such as steam distillation are traditionally used, due to their low cost. The advantage of this technique is the immiscibility between the organic compounds and water, facilitating the removal of the product,

however, the high temperatures used can generate changes in the oil obtained [8-10]. Another technique for obtaining the volatile extract is the supercritical extraction using CO₂, which preserves the properties of thermosensitive compounds, performing their extraction with minimal damage, in addition to the easy removal of the solvent at the end of the extraction. However, it is still a technique with a high implementation cost due to the high pressures used [11-12].

The different extraction techniques available generate products with differences in their chemical composition, making it necessary to determine the different compounds obtained from each type of extraction [13]. For this, it is necessary to use multivariate statistical analysis, such as, for example,

the principal components analysis, which allows an analysis that would not be easily achieved with the simple study of the data, due to its ability to reduce variables. Thus, it is possible to verify the variation in the composition, according to the extraction method and the condition used.

Due to the lack of technology associated with the essential oils industry, it is necessary to include scientific data to meet this demand. Therefore, the adjustment of mathematical models that adequately represent the yield versus time extraction curve, as well as obtaining mass transfer parameters, is an important step for the proper scale-up of extraction processes. Among the different types of models used to describe extraction processes, a first-order model can be highlighted, extensively used in adsorption processes [14], diffusion models [15], and models that describe differential balances for each phase [16].

The objective of this work is to carry out a comparison between the steam distillation technique and supercritical extraction, evaluating in terms of yield and composition (variation according to the technique and condition employed), as well as through mathematical modeling of the processes to analyze the representativeness of the model for each extraction method.

2. Materials and methods

2.1 Plant preparation

The plant material used in this study is composed of turmeric rhizomes grown in Morrinhos do Sul and purchased in Porto Alegre – Rio Grande do Sul, Brazil (-29.30, -49.92).

The turmeric rhizomes in natura were cut into irregular pieces, without removing the peel. The wet plant was ground using a knife mill and a sample was collected to quantify the moisture, using a thermogravimetric balance at 60 °C. The fresh grounded material was then used to carry out the experiments.

2.2 Extraction with supercritical fluid

The supercritical fluid extractions (SFE) were carried out in a pilot scale equipment, installed in the Laboratory of Unit Operations (LOPE) of the Polytechnic School – PUCRS. The system is represented in a schematic diagram in Fig. 1. The pilot unit has a high-pressure pump (Maximator-G60) for

carbon dioxide (P1), a CO₂ storage cylinder (C1), two preheaters (HE1, HE2), a system for measuring the CO₂ flow and two separation vessels (VS1, VS2), which are made of glass (Ilmaból TGI Boron 3.3). The extractions with supercritical fluid were carried out in an extraction container (Waters) with a capacity of 500 mL, diameter of 6.3 cm, and height of 19 cm [17]. The investigated conditions were determined according to Scopel et al. [18] and Garcez et al. [19], in order to obtain the volatile extracts using the SFE methodology: four different pressures (80, 90, 100, and 110 bar) at 40 °C, with 1000 g.h⁻¹ of CO₂ flow and 0.2 kg of the rhizome. Bearing in mind that the composition of the essential oil of *Curcuma longa* has in its majority composition polar compounds [20-22], the wet plant was used in the extractions with supercritical CO₂, as the presence of water would confer polarity to the solvent, favoring the extraction of these major components. This procedure is similar to the one used for caffeine extraction from coffee by humidifying the grain [23].

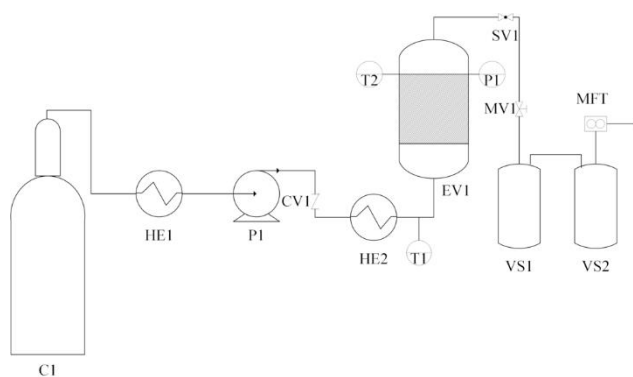


Figure 1. Diagram of the supercritical extraction pilot unit: C–CO₂ cylinder, HE – heat exchanger, CV– check valve, P1–CO₂ high pressure pump, EV–extraction vessel, T–temperature transmitter, P–pressure transmitter, VS–separation vessel, MFT–mass flow transmitter, SV– stop valve.

For the highest yield condition, the extract yield versus time curve was constructed in triplicate, for subsequent mathematical modeling. The extracts were collected with a time interval of 10 minutes, measuring mass at each time interval, until the plant was exhausted, that is until there was no mass increase (constant mass was considered after three consecutive measurements). The yield was obtained by dividing the extracted mass by the plant mass used in the extraction.

2.3 Steam distillation

The extractions via steam distillation (SD) were carried out in a pilot scale equipment, located in the Laboratory of Unit Operations (LOPE) of the Polytechnic School–PUCRS. The system is represented in a schematic diagram, as shown in Fig. 2. The equipment has a boiler (B1) with a capacity of 20 L of solvent (water), the energy source is an electrical resistance of 2 kW, it has level sensors (upper and lower) and pressure and temperature measurement. The extraction vessel (EV1) has a capacity of 10 L of useful volume, as well as temperature and pressure sensors, is 31.3 cm high and 19.3 cm in diameter. The system also has a multitubular shell-tube condenser cooled with water close to 1°C, using a thermostatic bath [24].

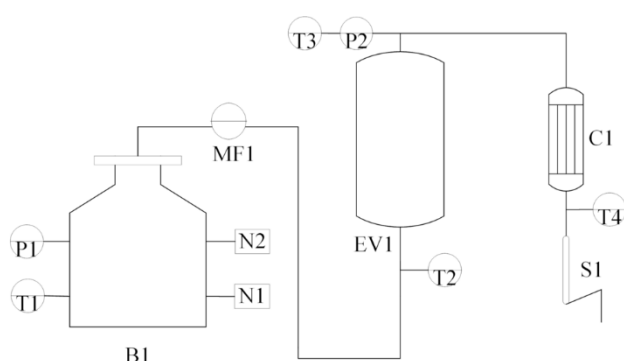


Figure 2. Steam distillation pilot plant schematic: B – boiler, EV – extraction vessel, C – condenser, S – separator, T – temperature transmitter, P – pressure transmitter, MF – flow meter, N – flow measurement level.

The steam distillation process was carried out with 1000 g of the prepared plant. The extractions were carried out at three different absolute pressures (1, 2, and 3 bar), with the aim of analyzing the effect on the yield and composition of the essential oil. The procedure was performed in triplicate, and for the highest yield condition, the experimental yield curve versus the extraction time was constructed, measuring the oil volume for each 5 minutes' interval.

The specific mass of the essential oil was determined by measuring the mass of 1 mL of the oil, using an analytical balance (Mars AW220 e = ± 0.0001g). This procedure was performed in triplicate. Plant density and porosity were determined using a pycnometer (Quantachrome MVP-6DL).

2.4 Chromatographic Analysis via GC-MS

Turmeric extracts were dehydrated with anhydrous

sodium sulfate (Na_2SO_4 - Synth) and diluted in cyclohexane (1:2) (Merck). Chemical composition was determined using a gas chromatograph equipped with a mass spectrometer (Hewlett Packard and Agilent model 7890A CG-EM and mass detector 5975C). The carrier gas was helium ($0.8 \text{ mL}\cdot\text{min}^{-1}$), the injector temperature was 250 °C, and the injected volume was 0.2 μL , using split mode with a division rate of 1:55. The capillary column was HP-5MS (Hewlett Packard and Agilent, 5% phenyl methyl siloxane, 30 m 250 mm 0.25 mm). The temperature setting was 60 °C (8 min), 60 °C-180 °C, 3 °C min^{-1} , 180 °C (1 min), 180 °C-250 °C, 20 °C min^{-1} , 250 °C (10 min).

Components were identified by comparing their Retention Index (RI) on the column, determined in relation to a homologous series of n-alkanes (C8-C20), with those of pure standards or reported in the literature. A comparison of the mass spectra of the compounds with the spectra stored in the GC-MS database [25] was also performed.

2.5 Principal Component Analysis (PCA)

The comparative statistical analysis of the composition of the products obtained was carried out through the analysis of principal components which was realized using the compounds percentages obtained through gas chromatography, for each of the techniques and conditions. The PCA was executed in the Minitab® software, where the data were implemented in the form of a table, where the lines were the percentage of the components and the columns were the pressure conditions under which the extractions were performed, the calculation was performed from the covariance of data.

2.6 Mathematical Modeling

The model used in this work was based on the model developed by Reverchon [16]. The model consists of a one-dimensional mass balance for the extract, assuming a linear behavior for the solid-fluid phase equilibrium. Two independent variables, time (t) and fixed bed height (z) were considered only, and the radial dispersion along the column is considered insignificant, for these assumptions the model was developed. The mass balance is given below in Equations 1 and 2.

Fluid phase mass balance:

$$\frac{\partial C(z,t)}{\partial t} = -v \frac{\partial C(z,t)}{\partial z} - \frac{1-\varepsilon}{\varepsilon} \rho_s \frac{\partial q(z,t)}{\partial t} \quad (1)$$

Mass balance in the solid phase:

$$\frac{\partial q(z,t)}{\partial t} = -k_{TM}[q(z,t) - K \cdot C(z,t)] \quad (2)$$

The concentration of the essential oil in the vapor phase is given by the function $C(z,t)$ and the concentration in the aromatic plant is described by the function $q(z,t)$. Where v is the interstitial fluid velocity; ε is the porosity of the bed; k_{TM} is the internal mass transfer coefficient; ρ_s is the specific mass of the plant and K is the equilibrium constant between phases. The model also considers some initial and boundary conditions $q(z,0) = q_0$ and $C(z,0) = 0$, q_0 is defined by the total amount of extract contained in the solid phase and $C(z,0) = 0$ as a boundary condition. The linear compartment for solid-fluid phase equilibrium is expressed by $q^*(z,t) = K \cdot C(z,t)$.

The described models were programmed in the EMSO software, where the parameters of each model were estimated by adjusting the experimental data of the extraction curve by the least squares method, using the Nelder-Mead algorithm [26].

3. Results and discussion

The average yield obtained from the experimental data acquired through the extractions is shown in Table 1 in essential oil grams per 100 grams of plant. The average specific mass of the essential oil obtained was $Q_{oil} = 890 \text{ kg.m}^{-3}$, the plant specific mass determined using the pycnometer was $Q_{plant} = 1087 \text{ kg.m}^{-3}$, a moisture of 80.4% were determined and the plant particle diameter was equal to 1.45 mm.

3.1 Analysis of the turmeric essential oil.

The primary constituents of turmeric essential obtained via SFE were α -zingiberene (22.78-10.16 %), α -turmerone (24.12-11.38 %) and ar-turmerone (22.48-10.8 %). These results are in accordance with what was found by Carvalho et al. [27] but with lower content of β -turmerone and α -turmerone. These authors did not report the higher levels of α -zingiberene found in this work. The SD method showed that the major compounds were α -zingiberene (35.42-23.11 %), α -turmerone (24.52-20.24 %) and β -sesquiphelandrene (17.95-13.21 %).

Table 1. Yield obtained through SFE and SD

Extraction method	Pressure (bar)	Global yield (g _{oil} /100g _{plant})
SD ^a	1	0.78
	2	0.83
	3	0.86
SFE ^b	80	0.07
	90	0.89
	100	0.21
	110	0.04

a = saturated water vapor, b = fluid temperature 40 °C

This is consistent with the results of Hwang et al. [28], although with lower levels of ar-turmerone. These variations could be attributed to geographic location, genetic and environmental factors, as reviewed in Ibáñez [29]. Table 2 shows the compounds found in the essential oil obtained using different techniques and conditions, with their respective retention index and area percentage. The major compounds for each extraction method are highlighted.

3.2 Principal Component Analysis

From the PCA, it was possible to observe that five components stood out from the others, α -turmerone, ar-turmerone, β -turmerone, β -sesquiphelandrene, and α -zingiberene as shown in Fig. 3.

In Fig. 3, it is possible to observe the behavior of the composition of *Curcuma longa* essential oil related to the pressure variation and the extraction method, it can be noted the formation of two distinct groups. The first one demonstrates higher scores on the first component, with zero or negative scores on the second component. The second group, on the other hand, exhibits higher scores on the second component, but lower scores on the first component. This division of groups is attributed to the similarity of the area percentage of the components identified by GC-MS. It was possible to visualize that there was difference in the composition due to the different pressures used in the steam extraction. turmerone and β -sesquiphelandrene content, while at 1 and 2 bar the composition was similar.

For the supercritical extraction technique, it was possible to observe a variation in the composition obtained between the different pressures. The similarity between the pressures of 80 and 110 bar was observed, while 100 and 90 bar pressure composition

Table 2. Chemical composition of *Curcuma longa* essential oil obtained by supercritical extraction and steam distillation under different extraction conditions.

Compounds ^a	RI ^b	Steam Distillation Area (%) ^c				Supercritical Fluid Extraction Area (%) ^c		
		1 bar	2 bar	3 bar	80 bar	90 bar	100 bar	110 bar
1,8-Cineole	1028	-	0.56	-	0.13	-	-	-
Terpinolene	1085	1.28	1.13	-	0.07	-	-	-
3Z-hexenilmetil carbonate	1100	-	-	-	0.03	-	-	-
2-Nonanol	1101	-	-	-	0.09	-	-	-
Terpinen-4-ol	1174	-	-	-	0.05	-	-	-
1,4-Cymene-8-ol	1184	-	-	-	0.16	-	-	-
α -Terpineol	1188	-	-	-	0.27	-	-	-
2-Decanol	1202	-	-	-	0.02	-	-	-
2-Undecanol	1301	-	-	-	0.03	-	-	-
δ -Elemene	1335	-	-	-	0.03	-	-	-
Piperitenone	1338	-	-	-	0.13	-	-	-
β -Elemene	1389	-	0.48	-	0.12	-	-	0.07
Sesquijujene	1404	-	-	-	0.09	-	-	-
E-Caryophyllene	1415	-	0.34	-	0.14	-	-	0.08
γ -Elemene	1431	-	-	-	0.03	-	-	-
E- α -Bergamotene	1433	-	-	-	0.04	-	-	-
E- β -Farnesene	1456	-	0.54	-	-	-	0.41	-
Sesquisabinene	1456	-	-	-	-	-	-	0.23
Z- β -Farnesene	1456	-	-	-	0.32	-	-	-
γ -Amorphene	1477	-	-	-	-	-	-	0.08
Germacrene D	1477	-	0.42	-	0.09	-	-	-
ar-Curcumene	1481	4.02	3.6	5.17	1.49	1.45	3.31	1.79
α-Zingiberene	1494	25.26	23.11	35.42	11.37	17.44	22.78	10.16
β -Curcumene	1502	-	-	-	0.09	-	-	-
β -Bisabolene	1507	2.6	2.53	-	1.41	1.63	2.52	1.31
β-Sesquiphelandrene	1522	14.41	13.21	17.95	6.65	8.85	12.58	6.41
E- γ -Bisabolene	1530	-	0.27	-	0.19	-	-	0.12
E-iso- γ -Bisabolene	1534	-	-	-	0.09	-	-	-
γ -Cuprenene	1543	-	-	-	0.05	-	-	-
Germacrene B	1553	-	0.49	-	-	-	0.55	-
E-Nerolidol	1563	-	-	-	0.06	-	-	0.04
ar-Tumerol	1579	-	0.66	-	0.26	-	-	0.38
E- β -Elemenone	1602	-	-	-	0.36	-	-	0.49
β -Atlantol	1698	1.08	2.22	-	-	-	-	-
ar-Turmerone	1664	11.33	9.76	10.48	22.07	10.8	12.43	22.48
α-Turmerone^d	1673	24.00	20.24	24.52	5.32	24.12	18.57	11.38
Helifolenol A	1687	-	-	-	0.12	-	-	-
Germacrene	1692	4.36	4.52	-	2.77	3.36	2.86	3.76
β-Turmerone^d	1703	8.25	7.11	6.45	7.67	8.29	7.66	8.98
Curcuphenol	1719	-	-	-	0.25	-	-	0.44
Curcumenol	1727	-	-	-	0.85	-	-	0.75
6S,7R-Bisabolone	1745	1.53	1.73	-	1.84	1.6	-	2.27
6R,7R-Bisabolone	1754	-	-	-	-	-	1.31	-
β -Bisabolenal	1770	-	-	-	0.56	-	-	-
E- α -Atlantone	1775	0.83	1.38	-	0.21	-	-	0.25
Hexadecanoic acid	1973	-	-	-	0.05	-	-	-
dehydro-Juvibione	1997	-	-	-	0.08	-	-	-
Total identified	-	98.95	94.3	100	65.65	77.54	84.98	71.47

^aCompounds identified by comparing their mass spectrum and retention index with the Adams library (2007). ^bRI retention index calculated for a series of alkanes (C8-C20). ^cPercentage area of each peak, according to the response of the mass detector, in relation to the total area of the chromatogram, considering a response factor equal to 1 for all components. ^dCompounds identified by comparing their mass spectrum and retention index based on NIST library (2005).

come closer to the SD method but with a lower first component score, mainly due to the lower α -zingiberene and β -sesquiphelandrene content. The first and second components explain 98.3% of the data variance.

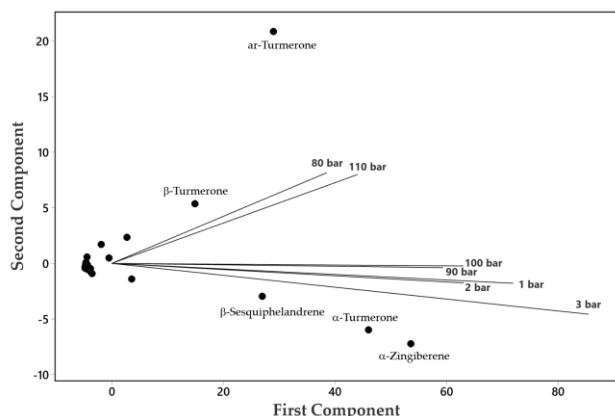


Figure 3. Variation in the behavior of turmeric essential oil, in relation to different pressures and extraction methods.

SD extract presented higher concentrations of α -zingiberene, which can indicate that its extract could be more effective in medicine in the treatment of some types of human cancer (liver [30], colon [31], cervical, and breast cancers and leukemia [32]) as well as an antibacterial agent for food industry [33]. The α -zingiberene compound is also potential insecticide and can induce resistance to diseases and pests in tomatoes [34-35]. The SFE extract presented a higher concentration of ar-turmerone which has a promising anticancer capacity, as cited before [7].

3.3 Mathematical Modeling

According to the proposed methodology, mathematical modeling was carried out and the results can be seen in Fig. 4. The modeling was realized for the highest yield conditions (3 bar for SD and 90 bar for SFE). The estimated values for the parameters, together with the coefficient of determination (R^2) for each method are presented in Table 3. The standard deviation for the yield of SD curve was $\pm 0.1194\%$ and for the SFE it was $\pm 0.1438\%$.

Table 3. Parameters obtained through modeling of the experimental data for SD and SFE methods.

Method	$K \cdot 10^4 \text{ m}^3 \cdot \text{kg}^{-1}$	$k_{TM} \cdot 10^4 \cdot \text{s}^{-1}$	R^2
SFE	12.403	1.7915	0.9931
SD – Stage 1	0.9975	1.0671	0.9739
SD – Stage 2	1.1213	1.3905	0.9888

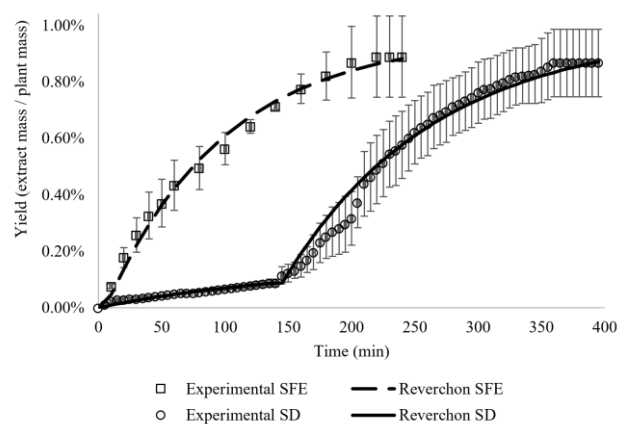


Figure 4. Curves for SFE (90 bar and 313.15 K) and SD (3 bar and 406.15 K) and mathematical modeling.

The best yield for supercritical extraction was at the condition of 90 bar and 40 °C. This is a condition close to the critical point where it is known that carbon dioxide has a higher density, which increases the solvent solubility. With the increase in pressure, maintaining a fixed temperature of 40 °C, it is known that the CO_2 density and diffusivity compete with each other. As the diffusivity decreases, the density increases, as both affect the solvation capacity of supercritical carbon dioxide, it passes through a maximum condition depending on which variable is prevailing in the considered condition [36-37].

From the SD extraction curve, it was possible to identify the presence of two different regimes. The first regime was identified as a curve of low slope followed by the second regime that ends with the depletion of the plant. This difference in mass transfer between these different periods for extraction can be explained by a change in the type of solute being removed, there are volatile compounds that are preferentially removed in an initial step. Afterwards, the matrix continues to provide extract mass, but this last is composed of different chemical species. With this, the mass balance for two pseudo components was considered, one leaving in the first stage and the other in the second stage. Thus, it was necessary to estimate the mass transfer parameters for each of these regimes. The regime change time was estimated based on the graphical analysis, occurring within 140 minutes.

The coefficient of determination corresponds to the adherence of the model to the experimental data. For

both extractions, the coefficient indicates that the model is adequate to fit the experimental data.

The K parameter is the equilibrium constant between the phases, which proved to be greater for the SFE, indicating that the equilibrium is predominant in the extraction compared to the SD, which obtained a lower order equilibrium constant. The k_{TM} parameter, which indicates mass transfer, had the same magnitude for both techniques. It is also worth mentioning that the SFE method could achieve 0.8% of yield way faster than SD method, being more time efficient than SD. The parameters obtained are in the expected order of magnitude, for SFE and SD [38].

4. Conclusions

It was possible to evaluate the performance of the SD technique and SFE, with higher yields being obtained at pressures of 3 bar and 90 bar, respectively. The chromatographic analysis of the extracts showed α -zingiberene as the major compound for the SD technique and ar-turmerone and α -turmerone for the SFE. From the PCA it was observed that the pressure did not significantly influence the composition of the essential oil for the SD. For the SFE, a similarity was identified between the composition of 90 bar and 110 bar, with the influence of the pressure on the extract composition. The mathematical modeling of the processes was carried out and the relevant parameters were obtained, with the SD technique composed of two periods. It was observed that the SFE was greater influenced by the process equilibrium in relation to the SD technique. It is possible to indicate that SD extract is more indicated for applications that need α -zingiberene and SFE extract for uses that request higher concentrations of ar-turmerone. The data obtained from the mathematical modeling support for the studies of change from a pilot scale to an industrial scale. In future work, the chemical composition of the extract will be monitored throughout the extraction, in order to validate the hypothesis of extraction of two types of solute and to help determine the parameters of the mass transfer model used for the steam distillation.

Authors' contributions

Conceptualization, E.T.S.J.; R.N.A.; E.C. and R.M.F.V.; Methodology, E.T.S.J. and A.M. L.; Formal Analysis, E.T.S.J.; R.N.A. and A.M.L.; Data Curation, E.T.S.J.;

Writing – Original Draft Preparation, E.T.S.J.; Writing–Review & Editing, A.M.L.; Supervision, R.N.A.; A.M.L.; E.C. and R.M.F.V.; Funding Acquisition, E.C. and R.M.F.V.

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Conflicts of interest

The authors declare that they have no known competing financial interests of or personal relationships that could have appeared to influence the work reported in this paper.

References

1. Custódio, H.N. Study of the extraction process of volatile and fixed fractions of turmeric oleoresin (*Curcuma longa* L.). Master Thesis, Goiás Federal University, Goiânia, Brazil, 2014.
2. LI, S.; Yuan, W.; Deng, G.; Wang, P.; Yang, P.; Aggarwal, B. Chemical Composition and Product Quality Control of Turmeric (*Curcuma longa* L.). Pharm. Crops 2011, 2, 28-54.
3. Ferreira, F.D.; Kimmelmeier, C.; Arrotéia, C.C.; Costa, C.L.; Mallmann, C.A.; Janeiro, V.; Ferreira, F.M.D.; Miossini, S.A.G.; Silva, E.L.; Jr. Machinski, M. Inhibitory effect of the essential oil of *Curcuma longa* L. and curcumin on aflatoxin production by *Aspergillus flavus* Link. Food Chem. 2013, 136, 789-793.
4. Avanço, G.B.; Ferreira, F.D.; Bomfim, N.S., Santos, P.A.S.R.; Peralta, R.M.; Brugnari, T.; Mallmann, C.A., Filho, B.A.A.; Mikcha, J.M.G.; Jr. Machinski, M. *Curcuma longa* L. essential oil composition, antioxidant effect, and effect on *Fusarium verticillioides* and fumonisin production. Food Control. 2017, 73, 806-813.
5. Saju, K.A.; Venugopal, M. N.; Mathew, M. J. Antifungal and insect-repellent activities of essential oil of turmeric (*Curcuma longa* L.). Curr. Sci. 1998, 75, 660-662.
6. Liju, V.B.; Jeena, K.; Kuttan, R. An evaluation of antioxidant, anti-inflammatory, and antinociceptive activities of essential oil from *Curcuma longa* L. Indian J. Pharmacol. 2011, 43, 526-531.
7. Nair, A.; Amalraj, A.; Jacob, J.; Kunnumakkara, A.B.; Gopi, S. Non-Curcuminoids from Turmeric and Their Potential in Cancer Therapy and Anticancer Drug Delivery Formulations. Biomol. 2019, 9, 1-36.

8. Takahashi, M. Pink pepper (*Schinus terebinthifolius* Raddi) essential oils and oleoresin: chemical and biological properties. Ph.D. Thesis, Federal University of Pelotas, Pelotas, Brazil, 2012.
9. Sartor, R.B. Modeling, simulation, and optimization of an industrial unit for the extraction of essential oils by steam distillation. Master Thesis, Federal University of Rio Grande do Sul, Porto Alegre, Brazil, 2009.
10. Cassel, E.; Vargas, R.M.F. Experiments and Modeling of the *Cymbopogon winterianus* Essential Oil Extraction by Steam Distillation. J. Mex. Chem. Soc. 2006, 50, 126-129.
11. Santos, D.N. Extraction with supercritical carbon dioxide and study of the composition of Pitanga (*Eugenia uniflora* L.) seed extracts. Master Thesis, University of São Paulo, São Paulo, Brazil, 2012.
12. Maia, J. D.; Alicieo, T.V.R. Supercritical Extraction Applied to Fruits – Review. Rev. Inova Ciênc. Tecnol. 2017, 3, 36-48.
13. Cassel, E. et al. Steam distillation modeling for essential oil extraction process. Ind. Crops Prod. 2009, 29, 171-176.
14. Dagostin, J.L.A.; Carpiné, D.; Corazza, M.L. Extraction of soybean oil using ethanol and mixtures with alkyl esters (biodiesel) as co-solvent: Kinetics and thermodynamics. Ind. Crops Prod. 2015, 74, 69-75.
15. Crank, J. The Mathematics of Diffusion, 2nd ed.; Bristol: Oxford University Press, UK, 1975.
16. Reverchon, E. Mathematical modeling of supercritical extraction of sage oil. AIChE Journal 1996, 42, 1765-1771.
17. Scopel, R., Góes Neto, R., Falcão, M.A., Cassel, E., Vargas, R.M.F. Supercritical CO₂ extraction of *Schinus molle* L with co-solvents: mathematical modeling and antimicrobial applications. Braz. Arch. Biol. Technol. 2013, 56, 513-519.
18. Scopel, R.; Silva, C.F.; Lucas, A.M.; Garcez, J.J.; Espírito Santo, A.T.; Almeida, R.N.; Cassel, E.; Vargas, R.M.F. Fluid phase equilibria and mass transfer studies applied to supercritical fluid extraction of *illicium verum* volatile oil. Fluid Phase Equilib. 2016, 417, 203-211.
19. Garcez, J.J.; Barros, F.; Lucas, A.M.; Xavier, V.B.; Fianco, A.L.; Cassel, E.; Vargas, R.M.F. Evaluation and mathematical modeling of processing variables for a supercritical fluid extraction of aromatic compounds from *Anethum graveolens*. Ind. Crops Prod. 2017, 95, 733-741.
20. Chassagnez-Méndez, A.L.; Machado, N.T.; Araujo, M.E.; Maia, J.G.; Meireles, M.A.A. Supercritical CO₂ Extraction of Curcumins and Essential Oil from the Rhizomes of Turmeric (*Curcuma longa* L.). Ind. Eng. Chem. Res. 2000, 39, 4729-4733.
21. Gopalan, B.; Goto, M.; Kodama, A.; Hirose, T. Supercritical Carbon Dioxide Extraction of Turmeric (*Curcuma longa*). J. Agric. Food Chem. 2000, 48, 2189-2192.
22. Priyanka; Khanam, S. Influence of operating parameters on supercritical fluid extraction of essential oil from turmeric root. J. Clean. Prod. 2018, 188, 816-824.
23. Franca, A.S. Coffee: decaffeination. Encycl. Food Health 2016, 232-236.
24. Souza Jr, E.T. De, Siqueira, L.M., Almeida, R.N., Lucas, A.M., Da Silva, C.G.F., Cassel, E., Vargas, R.M.F. Comparison of different extraction techniques of *Zingiber officinale* essential oil. Braz. Arch. Biol. Technol. 2020, 63.
25. Adams, R.P. Identification of essential oil components by gás chromatography / mass spectrometry, 4th ed.; Illinois: Allure, USA, 2007.
26. Nelder, J.A.; Mead, R.A Simplex Method for Function Minimization. The Comput. J. 1965, 7, 308-313.
27. Carvalho, P.I.N.; Osorio-Tobón, J.F.; Rostagno, M.A.; Petenate, A.J.; Meireles, M.A.A. Techno-economic evaluation of the extraction of turmeric (*Curcuma longa* L.) oil and ar-turmerone using supercritical carbon dioxide. J. Supercrit. Fluids 2015, 105, 44-54.
28. Hwang, K.W.; Son, D.; Jo, H.W.; Kim, C.H.; Seong, K.C.; Moon, J.K. Levels of curcuminoid and essential oil compositions in turmeric (*Curcuma longa* L.) grown in Korea. Appl. Biol. Chem. 2016, 59, 209-215.
29. Ibáñez, M.D.; Blázquez, M.A. *Curcuma longa* L. Rhizome Essential Oil from Extraction to Its Agri-Food Applications. A Review. Plants 2020, 10, 1-31.
30. Peng, X.; Luo, R.; Li, J.; He, A.; Wang, X.; Wan, H.; Cai, Y.; Dong, W.; Lin, J. Zingiberene targets the miR-16/cyclin-B1 axis to regulate the growth, migration and invasion of human liver cancer cells. JBUON 2020, 25, 1904-1910.
31. Chen, H.; Tang, X.; Liu, T.; Jing, L.; Wu, J. Zingiberene inhibits in vitro and in vivo human colon cancer cell growth via autophagy induction, suppression of PI3K/AKT/mTOR Pathway and caspase 2 deactivation. JBUON 2019, 24, 1470-1475.
32. Lee, Y. Cytotoxicity Evaluation of Essential Oil and its Component from *Zingiber officinale* Roscoe. Toxicol. Res. 2016, 32, 225-230.
33. Al-Dhahli, A.S.; Al-Hassani, F.A.; Alarjani, K.M.; Yehia, H.M.; Lawati, W.M.; Azmi, S.N.H.; Khan, S.A. Essential oil from the rhizomes of the Saudi and Chinese *Zingiber officinale* cultivars: comparison of chemical composition, antibacterial and molecular docking studies. J. King Saud Univ. Sci. 2020, 32, 3343-3350.
34. Oliveira, J.R.F.; Resende, J.T.V.; Lima Filho, R.B.; Roberto, S.R.; Silva, P.R.; Rech, C.; Nardi, C. Tomato Breeding for Sustainable Crop Systems: high levels of zingiberene providing resistance to multiple arthropods. Hort. 2020, 6, 34.

35. Zanin, D.S.; Resende, J.T.V.; Zeist, A.R.; Lima Filho, R.B.; Gabriel, A.; Diniz, F.C.P.; Perrud, A.C.; Morales, R.G.F. Selection of F2BC1 tomato genotypes for processing containing high levels of zingiberene and resistant to tomato pinworms. *Phytoparasitica* 2020, 49, 265-274.
36. Kopcak, U.; Mohamed, R.S. Caffeine solubility in supercritical carbon dioxide/co-solvent mixtures. *J. Supercrit. Fluids* 2005, 34, 209-214.
37. Taylor, L.T. *Supercritical fluid extraction*, vol. 4; Wiley-Interscience: New Jersey, USA, 1996.
38. Almeida, R.N.; Neto, R.G.; Barros, F.M.C.; Cassel, E.; Poser, G.L.; Vargas, R.M.F. Supercritical extraction of *Hypericum caprifoliatum* using carbon dioxide and ethanol + water as co-solvent. *Chem. Eng. Process.: Process Intensif.* 2013, 70, 95-102.