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Non-conventional techniques for the extraction of antioxidant compounds and lycopene from industrial tomato pomace (*Solanum lycopersicum* L.) using spouted bed drying as a pre-treatment

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ABSTRACT

This study aimed to use the non-conventional microwave-assisted extraction (MAE) and pressurized liquid extraction (PLE) techniques for recovering bioactive compounds from tomato pomace, a valuable agro-industrial waste. The raw material was previously dried using a spouted bed dryer and then submitted to extraction with green solvents. A response surface methodology (RSM) performed the optimization of MAE and PLE. Next, the yield and the antioxidant activity results were maximized, and the lycopene content of the optimum MAE and PLE extracts was assessed by high-performance liquid chromatography (HPLC). Additionally, a fraction of raw material was oven dried as a comparison. The PLE extract exhibited the highest antioxidant activity, whereas the MAE extract showed the highest lycopene content (59.66 μg lycopene/g extract), which represents a 66.93% lycopene recovery compared to a standard technique with acetone. The remarkable results show that the non-conventional drying and extraction techniques were effective in valorizing this neglected material.

1. Introduction

Tomato (*Solanum lycopersicum* L.) is a versatile and healthy food, which can be consumed in both raw or processed forms. Also, this fruit has several antioxidant compounds, such as carotenoids, flavonoids, phenols, ascorbic acid (vitamin C), and tocopherol (vitamin E) (Koh et al., 2012). Because of its limited shelf life and a short period of agricultural production, its most frequent consumption is as processed products (Koh et al., 2012).

A significant amount of fresh tomato is destined for processing into juice, pulps, ready-made sauces, canned peeled tomatoes, purees, ketchup, and soups (Koh et al., 2012). During industrial processing, a considerable amount of tomato pomace composed of skins, seeds, and vascular tissues is generated and represents about 4% of the total mass of the fruit. Currently, this pomace is wasted or used for animal feed production; nevertheless, this waste material is rich in nutrients and bioactive compounds (Del Valle et al., 2006; Hatami et al., 2019). Thus, in order to reduce economic losses and environmental impacts, several studies have been carried out to add value to this by-product (Naviglio et al., 2008; Nour et al., 2015; Scaglia et al., 2020; Silva et al., 2019; Zuorro et al., 2013).

When it comes to tomato nutrients and bioactive compounds, it is impossible not to mention the lycopene. This carotenoid accounts for 80–90% of all carotenoids in tomatoes and is enriched in red, yellow, or orange vegetables, fruits, and flowers (Liang et al., 2019). Once the content of lycopene and β-carotene in tomato pomace is around 400% higher than fresh tomatoes (Koh et al., 2012), the use of tomato pomace as the raw material for extracting lycopene is much more viable than the use of the fruit itself

The drying is a fundamental processing step for fruit by-products as it extends storage life, facilitates handling, reduces transportation costs, and improves further processing such as milling for taking far less energy than a wet product to be milled (Mujumdar, 2006). Within this framework, the process of drying in spouted beds has been a promising

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alternative for drying pasty materials such as tomato pomace, resulting in a powder with high quality and low cost. This technique is widely used in the dehydration of thermolabile materials as it preserves bioactive compounds and other structures of interest, thus presenting an advantage over other drying methods, especially when the processing time is taken into account (Bezerra et al., 2013).

In terms of spouted bed drying principles, the difference between drying pastes and solid particles is due to the presence of small inert particles which simultaneously serves as a support for the paste and a source of heat for drying (Epstein & Grace, 2010). A complete description of the fundamentals and applications of spouted bed drying can be found in the book by (Epstein & Grace, 2010).

Under the context of an integrated valorization of by-products, conventional extraction techniques are commonly applied to recover bioactive compounds from natural sources. These techniques are relatively simple but, their high time-consuming operation, requiring a large amount of organic solvent, cannot be overlooked (Chemat et al., 2020).

On the other hand, non-conventional extraction techniques are a less aggressive alternative to health and the environment, decrease the sample degradation and improve the extraction selectivity. These methods seek for the application of food-grade solvents such as ethanol and ethyl acetate, both recognized as GRAS (Generally Recognized as Safe) by Food & Drug Administration (FDA) (Danlami et al., 2014; U.S. Food & Drug Administration, 2020). Among these methods, Microwave-Assisted Extraction (MAE) and Pressurized Liquid Extraction (PLE) stand out for being recognized as green techniques, consuming less solvent, and having a shorter extraction time compared to conventional techniques (Mustafa & Turner, 2011; Vinatoru et al., 2017).

To the best of our knowledge, only a few studies have been published regarding the use of non-conventional techniques for obtaining tomato extracts, such as those by Ho et al (2015) and Pinela et al (2016) using MAE, Naviglio et al (2008) using pressurized water extraction, Scaglia et al (2020) using supercritical fluid extraction (SFE), and Lianfu & Zelong (2008) using ultrasound-assisted extraction. However, as far as we know, this is the first investigation in which tomato pomace was used as raw material for PLE and MAE, taking the antioxidant activity as response.

Therefore, the objective of this study was to obtain tomato pomace extracts with high antioxidant activity and high lycopene recovery using two non-conventional extraction techniques: MAE and PLE. For this, the following specific objectives were considered: (i) to submit the raw material to a previous non-conventional drying (spouted bed drying); (ii) to determine the optimum MAE and PLE conditions for maximizing the yield and the antioxidant activity using a Box-Behnken Design (BBD) with Response Surface Methodology (RSM); (iii) to verify the lycopene content of the optimum MAE and PLE by High-Performance Liquid Chromatography (HPLC).

2. Material and methods

2.1. Sample acquisition

The industrial tomato pomace composed of seeds and peels was kindly donated by the company Cargill (Goiânia, GO, Brazil). The raw material was stored in polyethylene bags and frozen at −18 °C until the drying procedures at the Laboratory of Thermo-fluid dynamics and Particulate Systems (LATESP) of the Federal University of Goiás (UFG).

2.2. Drying and determination of moisture content

2.2.1. Spouted bed drying

An illustration of the spouted bed dryer system used in the present study (Fig. S1), as well as the description of its parts and operation mode can be assessed in the Supplementary Material.

The drying conditions were set based on preliminary studies of LATESP research group. First, the bed was filled with 400 g of inert particles and 320 g of fresh tomato pomace. A bed height of 15 cm (with the aid of a U-tube manometer), a temperature of 70 °C, and a drying time of 1.5 h were defined to preserve the thermo-labile compounds. After drying, the sample was weighed and submitted to moisture analysis at 105 °C according to the gravimetric method (n° 925.10) described by the Association of Official Analytical Chemists (1997). Following this, the dry material was sent to the Laboratory of Thermodynamics and Supercritical Technology (LATESC) of the Department of Chemical and Food Engineering at the Federal University of Santa Catarina (EQA/UFSC).

2.2.2. Oven drying

A fraction of industrial tomato pomace was spread onto metallic trays and dried in an air circulation oven (De Leo, Porto Alegre, RS, Brazil) at 70 °C for 12 h. Next, the dry material was weighed, submitted to moisture analysis as described in section 2.2.1, and finally sent to LATESC.

Different from the spouted bed-dried, the oven-dried sample was only used as raw material for the optimized conditions in order to compare the efficiency of both drying techniques in terms of yield and in their capacity to preserve the lycopene and the antioxidant compounds from degradation.

2.3. Grinding

The dried samples were ground in a Willey knife mill (De Leo, Porto Alegre, RS, Brazil) and the particle size was determined by a vertical vibratory sieve shaker (Bertel Indústria Metalúrgica Ltda, Caieiras/SP, Brazil) according to the Association of Official Analytical Chemists (1997). Then, the average particle diameter was calculated using the equation presented by ASAE standard procedure (American Society of Agricultural and Biological Engineers, 2003). Finally, the samples were stored in opaque high-density polyethylene flasks (HDPE) at 4 °C until extractions in order to preserve the photosensitive compounds from degradation.

2.4. Extraction

2.4.1. Microwave-assisted extraction (MAE)

The MAE assays were carried out using a MonowaveTM 300 microwave (Anton Paar, Graz, Austria) with a constant solute to solvent ratio of 1:20 w/v. The specific MAE variables are described in section 2.7. Next, the solutions were filtered through qualitative filter paper (J. Prolab 80 g; diameter of 12.5 cm; pore opening of 14 μm; thickness of 205 μm) and submitted to rotary evaporation (Fisatom, Model 801, São Paulo, SP, Brazil) for the solvent removal. The dry extracts were stored in amber flasks at −18 °C until further analysis.

2.4.2. Pressurized liquid extraction (PLE)

The PLE assays were carried out in an extraction unit developed at LATESC and minutely described by Gonçalves Rodrigues et al (2019).

First, 5 g of sample was inserted into the 90 mL extraction vessel and its void fraction was filled with cotton layers and glass beads. Following this, the solvent was delivered to the system by an HPLC pump (Waters, Model 515, Milford, MA, USA), passed through a pre-heating vessel to reach the target temperature, and then flowed downstream through the extraction vessel. Once the extraction pressure of 100 bar was reached, the needle valve was opened and the extraction began in a continuous mode. During the extraction process, 100 mL of solvent were used, maintaining the same 1:20 w/v solute to solvent ratio used for MAE. The specific PLE variables can be seen in section 2.7.

Finally, the extracts were submitted to rotary evaporation and stored in amber flasks at −18 °C until further analysis.

2.4.3. Soxhlet

Both spouted bed- and oven-dried samples were submitted to a

continuous extraction by using the conventional Soxhlet method with ethanol (EtOH) and ethyl acetate (EA) as solvents per 6 h following the procedure 922.06 from the Association of Official Analytical Chemists (1997). Afterward, the solvents were eliminated using a rotary evaporator and the samples were stored in amber flasks at −18 °C until further analysis. The results were expressed as mean values of triplicate assays ± standard deviation.

2.5. Extraction yield

The global extraction yield (Equation (1)) was calculated as the ratio between the mass of dried extract (m_{ext}) and the mass of dried sample (m_{sample}).

$$Extractionyield(\%) = \frac{m_{ext}}{m_{sample}} \times 100 \quad (1)$$

2.6. Antioxidant activity

The antioxidant activity of tomato pomace extracts was assessed by the following methods: *2,2–Diphenyl-1-picrylhydrazil* (DPPH) method according to Brand-Williams et al (1995) and adapted for a 96-well plate; the ferric reducing antioxidant power (FRAP) method according to (Benzie & Strain, 1996) and adapted for a 96-well plate, and the β-Carotene bleaching method according to Matthäus (2002), with some modifications by Gonçalves Rodrigues et al (2019).

Both DPPH and FRAP results were expressed as μmol Trolox equivalent per g of extract (μmol TE/g) whereas β-carotene as % of antioxidant activity at 1.67 mg/mL extract concentration. All results were presented as the mean value of triplicate assays ± standard deviation.

The methodologies are covered in detail in the Supplementary Material.

2.7. Optimization of MAE and PLE using response surface methodology (RSM)

A Box-Behnken design (BBD) with response surface methodology (RSM) was applied to optimize the MAE and PLE conditions in terms of extraction yield, DPPH, FRAP, and β-Carotene bleaching method, coded as Y1, Y2, Y3, and Y4, respectively. For this purpose, the following factors were considered: temperature (T), solvent mixture ethanol (99.8%) and ethyl acetate (99.5%) (Neon, São Paulo, SP, Brazil) (E), and time (t) for MAE, and T, E, and solvent flow rate (F) for PLE, providing 15 experimental assays, with triplicate at the central point, for each extraction technique. Real and coded values of the factors are summarized in Tables 1 and 2, for MAE and PLE assays, respectively. The experimental data were fitted with the following second-order polynomial model (Equation (2)):

$$\beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{1.1} {X_1}^2 + \beta_{2.2} {X_2}^2 + \beta_{3.3} {X_3}^2 + \beta_{1.2} X_1 X_2 + \beta_{1.3} X_1 X_3 + \beta_{2.3} X_2 X_3 \quad (2)$$

where Y is the response variable (dependent), β_0 is the intercept, β_1, β_2, and β_3 are the linear coefficients, $\beta_{1.1}$,$\beta_{2.2}$, e $\beta_{3.3}$ are the quadratic coefficients, $\beta_{1.2}$,$\beta_{1.3}$ and $\beta_{2.3}$ are the interaction coefficients, X_1 is the independent variable T, X_2 is the independent variable E, and X_3 is the independent variable t (for MAE) and F (for PLE).

The effects of the independent variables (factors) on Y were evaluated by the Pareto charts, and the RMS illustrates the regions that maximize the responses.

Following this, the global optimum was attained by the application of the desirability function proposed by Derringer & Suich (1980). In the present study, the global optimum is the unique condition of T, E, and t (for MAE)/F (for PLE) that simultaneously maximizes all responses.

The software Statistica 13.5 (StatSoft, Tulsa, OK, USA) was used for the experimental design, data analysis, and evaluation of the global optimum.

2.8. Lycopene content

The lycopene fraction of the spouted bed-dried sample was obtained according to the method described by Fagundes et al (2015), as follows: two point five grams of the dried sample were mixed with 20 mL of acetone, with subsequent magnetic stirring for 1 h at 25 °C, in the absence of light. Following this, the solutions were filtered through a cellulose membrane under vacuum and transferred to centrifuge tubes. Next, 20 mL of petroleum ether and 10 mL of deionized water were added to the tubes and the samples were centrifuged at 1620 *g* for 10 min. Subsequently, the solution was transferred to a 50 mL volumetric flask, making the total volume with petroleum ether. Finally, the solvents were eliminated using a rotary evaporator. This first step consists of the total lycopene recovery.

The identification and quantification of lycopene from the extracts were performed according to Fagundes et al (2015). In sum, the optimum MAE and PLE extracts, as well as the Soxhlet extract and the lycopene fraction obtained with acetone were dissolved in 2.5 mL

Table 1
Box-Behnken (BBD) with real and coded independent variables (in parentheses), and response variables from the extraction of tomato pomace by microwave-assisted extraction (MAE).

Assay	Independent variables			Response variables			
	T (°C)	E (v/v)	t (min)	Y_1 Yield (%)	Y_2 DPPH (μmol TE/g)	Y_3 FRAP (μmol TE/g)	Y_4 β-Carotene (%)
1	50 (−1)	10:90 (−1)	3 (0)	3.83	8.63	13.02	22.40
2	90 (+1)	10:90 (−1)	3 (0)	5.40	14.05	18.52	36.19
3	50 (−1)	90:10 (+1)	3 (0)	3.95	9.69	15.93	33.96
4	90 (+1)	90:10 (+1)	3 (0)	6.42	15.06	24.52	39.82
5	50 (−1)	50:50 (0)	1 (−1)	4.50	8.85	12.13	22.84
6	90 (+1)	50:50 (0)	1 (−1)	6.64	11.92	14.50	27.60
7	50 (−1)	50:50 (0)	5 (+1)	4.76	10.39	14.76	24.30
8	90 (+1)	50:50 (0)	5 (+1)	6.16	13.17	15.84	32.47
9	70 (0)	10:90 (−1)	1 (−1)	4.73	11.11	15.78	26.78
10	70 (0)	90:10 (+1)	1 (−1)	4.84	12.78	18.45	32.31
11	70 (0)	10:90 (−1)	5 (+1)	4.92	12.54	17.99	32.20
12	70 (0)	90:10 (+1)	5 (+1)	5.27	13.49	22.27	34.78
13	70 (0)	50:50 (0)	3 (0)	5.08	11.58	14.11	26.53
14	70 (0)	50:50 (0)	3 (0)	5.32	11.12	13.33	27.60
15	70 (0)	50:50 (0)	3 (0)	5.42	11.28	13.39	28.40

T: temperature; E: solvent mixture ethanol:ethyl acetate; t: extraction time; DPPH: 2,2–Diphenyl-1-picrylhydrazil; FRAP: ferric reducing antioxidant power; β-Carotene: beta carotene bleaching method.

Table 2
Box-Behnken (BBD) with real and coded independent variables (in parentheses), and response variables from the extraction of tomato pomace by pressurized liquid extraction (PLE).

Assay	Independent variables			Response variables			
	T (°C)	E (v/v)	F (mL/min)	Y_1 Yield (%)	Y_2 DPPH (μmol TE/g)	Y_3 FRAP (μmol TE/g)	Y_4 β-Carotene (%)
1	50 (−1)	10:90 (−1)	5 (0)	7.71	13.32	6.15	23.31
2	90 (+1)	10:90 (−1)	5 (0)	9.83	18.33	25.51	40.23
3	50 (−1)	90:10 (+1)	5 (0)	11.76	10.19	5.86	19.70
4	90 (+1)	90:10 (+1)	5 (0)	16.61	14.91	16.82	26.69
5	50 (−1)	50:50 (0)	2 (−1)	12.62	11.56	9.23	31.15
6	90 (+1)	50:50 (0)	2 (−1)	15.18	19.10	22.01	46.51
7	50 (−1)	50:50 (0)	8 (+1)	11.31	11.56	5.64	37.76
8	90 (+1)	50:50 (0)	8 (+1)	13.32	13.36	8.34	39.65
9	70 (0)	10:90 (−1)	2 (−1)	11.34	14.98	20.02	44.77
10	70 (0)	90:10 (+1)	2 (−1)	15.73	13.22	14.39	30.90
11	70 (0)	10:90 (−1)	8 (+1)	9.84	13.36	18.51	40.23
12	70 (0)	90:10 (+1)	8 (+1)	15.63	10.17	13.67	27.02
13	70 (0)	50:50 (0)	5 (0)	11.85	11.45	21.71	25.92
14	70 (0)	50:50 (0)	5 (0)	11.44	9.87	20.69	28.45
15	70 (0)	50:50 (0)	5 (0)	11.49	9.97	20.97	28.76

T: temperature; E: solvent mixture ethanol:ethyl acetate; F: solvent flow rate; DPPH: 2,2–Diphenyl-1-picrylhydrazil; FRAP: ferric reducing antioxidant power; β-Carotene: beta carotene bleaching method.

hexane, and 10 μL of the solution formed were injected into a liquid chromatography (LC-10A, Shimadzu, São Paulo, SP, Brazil) equipped with reversed-phase C18 column (Vydac 218TP54, 250 × 4.6 mm, the internal diameter of 5 mm, 30 °C) and a UV–vis detector operating at 470 nm.

A methanol: acetonitrile solution (90:10, v/v) was used as a mobile phase with a flow rate of 0.8 mL/min, and the identification of lycopene was carried out by comparing the retention time of the peaks in the samples with the corresponding analytical standard (Sigma-Aldrich Chemie, Steinheim, Germany). The quantification of lycopene in the samples was conducted through a standard curve, and the results were expressed as μg lycopene/g extract ± standard deviation, resulting from the average calculation of three consecutive injections.

3. Results and discussion

3.1. Sample characterization

The tomato pomace had an initial moisture content of 74.3%, within the range of 70.0% to 95.0% found in the literature data (Al-Muhtaseb et al., 2010; Nour et al., 2015). After drying, the spouted bed- and the oven-dried samples showed moisture contents of 3.80 ± 0.20 and 4.60 ± 0.60% d.b. respectively. According to Lavelli et al (2013), the microbiological stability of tomato pomace can be guaranteed when the product has moisture levels below 15% d.b. (Water activity < 0.6), and in this sense, the products resulting from both drying techniques can be considered microbiologically stable.

The average particle diameter was 0.48 ± 0.01 for the spouted bed-dried sample and 0.51 ± 0.01 mm for the oven-dried sample. These values are within the range recommended by Belwal et al (2018), who state that the particle size must range from 100 μm to 2 mm so that there is a greater contact surface between sample and solvent, increasing the mass transfer rate, and hence the extraction yield.

3.2. Optimization of the non-conventional extraction techniques

The influence of temperature (T), solvent mixture ethanol:ethyl acetate (E), and time (t) for MAE, and T, E, and solvent flow rate (F) for PLE were evaluated in order to achieve the optimal global condition that simultaneously maximizes all responses. Temperature and time were idealized to determine a condition that would not degrade the compounds present in the tomato pomace, and the solvents were chosen to obtain extracts in the light of the green chemistry concept, that is, using Generally Recognized as Safe (GRAS) solvents, such as ethanol and ethyl acetate. Moreover, the solvent mixture was adopted because the use of two solvents together can leverage the extraction yield (Mustafa & Turner, 2011). According to Pinela et al (2016), fitting mathematical models to the selected responses is essential to understand how accurately the RSM model can predict the ideal extraction conditions. In this context, the models for each response were built by fitting the second-order polynomial model of Eq (2) to the experimental values from MAE (Table 1) and PLE assays (Table 2). The significance of the coefficients was assessed using analysis of variance (ANOVA). Tables S1 to S8 of the Supplementary Material present the ANOVA results, as well as the values of the significant coefficients used for building the models.

3.2.1. MAE

3.2.1.1. Extraction yield (%). The mathematical model presented $R^2 = 0.89$, in addition to a lack of fit > 0.05 (Table S1), indicating an adequate fitting to the experimental data. By the Pareto chart (Fig. 1a), it can be seen that the linear T was the parameter of greatest influence in terms of yield, followed by the quadratic E, with a positive sign. Such observations show that the increase in temperature, within the established levels and keeping E constant, significantly increased the extraction yield, as seen in Table 1 and illustrated in Fig. 1a.

In addition, Fig. 1a illustrates that the increase in ethanol fraction favored the yield to a certain extent, around intermediate values. From this limit, the surplus increase of E significantly decreased the yield.

The results differ from those previously reported by Pinela et al (2016), who investigated the extraction of phenolic compounds and flavonoids from lyophilized tomato fruits by MAE. The authors obtained an extraction yield of ≈35% with pure ethanol, which could be justified by the higher temperature (180 °C) and process time (15 min) than those applied in the present study.

3.2.1.2. Antioxidant activity. All the models were adequately fitted with $R^2 > 0.88$, besides non-significant lack of fit ($p > 0.05$).

For the DPPH, the Pareto chart (Fig. 1b) indicates that the three independent variables had a significant influence on the response, mainly for linear T, and E^2, as observed for yield (section 3.2.1.1). However, the response surface shows that the influence of E^2 was distinct, i.e., once intermediate regions of E favored the yield, the application of pure solvents led to higher DPPH values. This finding emphasizes that, although the equivolumetric mixture of ethanol and ethyl acetate has favored the results in quantitative terms, this mixture impaired the results in qualitative terms.

The influence of the solvent was even more significant in the FRAP method, where the E^2 effect surpassed the effect of linear T, as seen in Fig. 1c. The response surface illustrates that, as with DPPH, the increase in temperature and, in particular, extreme values of E raised the response.

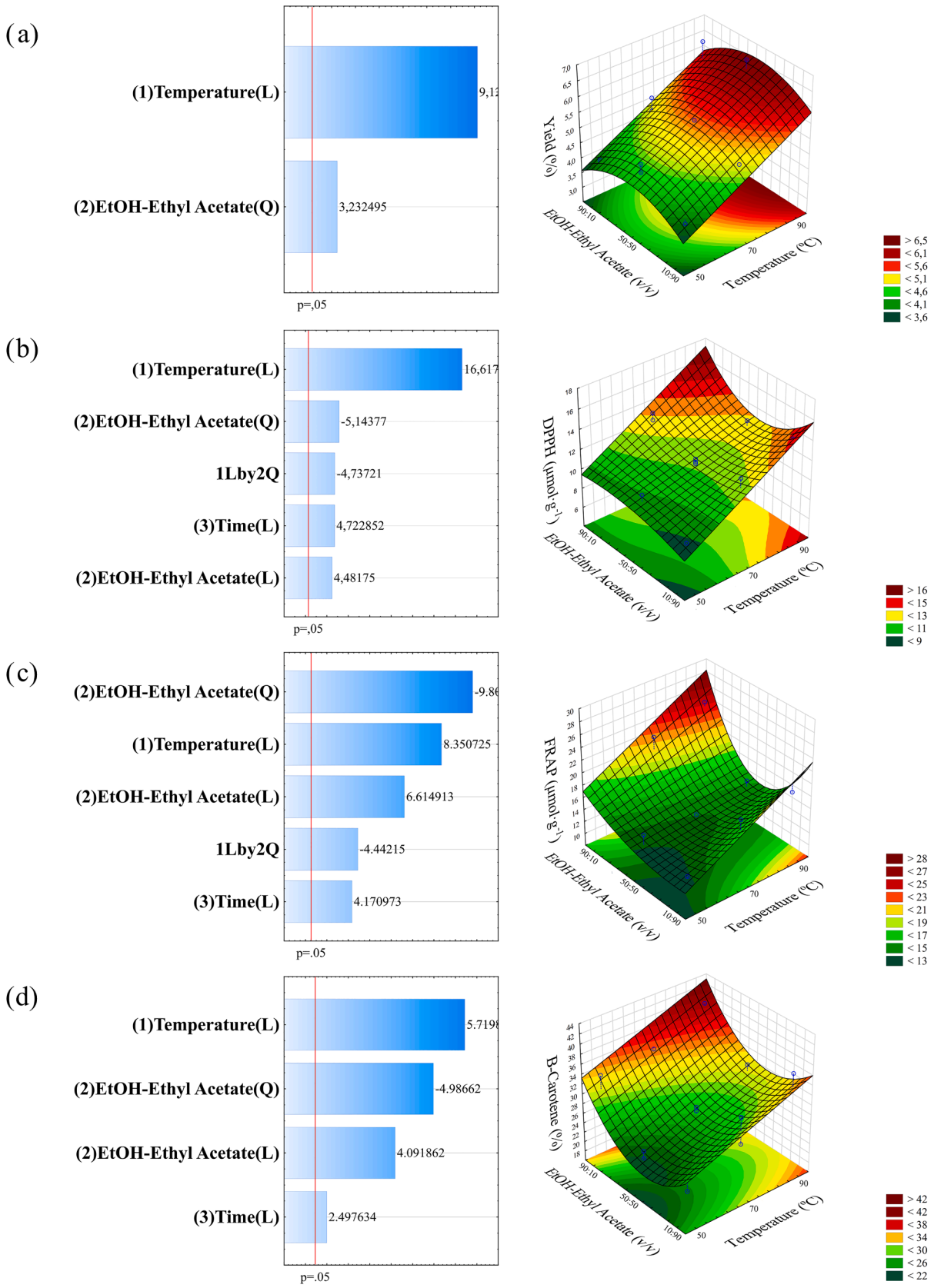

Fig. 1. Pareto charts for the response variables yield (1a), DPPH (1b), FRAP (1c), and β-Carotene (1d) studied in BBD of MAE, and their correspondent response surfaces.

For the β-Carotene bleaching method, all factors were statistically significant, with emphasis on T and E^2, as shown in Fig. 1d. In addition, the same behavior obtained for DPPH and FRAP can be noted: higher antioxidant activity found at the highest temperature and toward pure solvents, with a greater predilection for the ethanol, which was also observed for the other antioxidant methods.

According to Kappe et al (2008), the efficiency of the MAE depends on the dissipation factor (tan δ) of the solvent, its ability to absorb microwave energy and transmit it to the matrix as heat. The solvents can be classified as good (tan $\delta > 1$), medium (tan $\delta = 0.1$–0.5) and low (tan $\delta < 0.1$) microwave absorbers. The tan δ value of ethanol is 0.941, being considered as an excellent absorber, whereas the tan δ value of ethyl acetate is 0.059, which makes it a low absorber (Kappe et al., 2008). This could explain the fact that the highest values of antioxidant activity were obtained at higher fractions of ethanol, which corroborates previous results related to MAE of tomato samples reported by Pinela et al (2016).

Moreover, the temperature effect was also significant, once the temperature rise led to higher antioxidant activity. Similar behavior was observed by Dewanto et al (2002), where the authors reported an increase in the antioxidant activity of heat-processed tomatoes (88 °C per 2, 5, and 30 min) compared to the raw sample, despite the decrease in Vitamin C content. According to the authors, although high temperature favors the disruption of cell walls, releasing oxidative and hydrolytic enzymes that can destroy antioxidants in fruits and vegetables, the thermal processing at 88 °C deactivated these enzymes, preserving phenolic acids, compounds that exert antioxidant activity in tomatoes.

Based on the mathematical models and the Pareto charts: i) the influence of the extraction parameters was more significant in antioxidant activity than in extraction yield; ii) as for yield, T and E remained the most significant factors.

3.2.2. PLE

3.2.2.1. Extraction yield. The fitted model exhibited a non-significant lack of fit ($p > 0.05$) besides a high $R^2 = 0.98$, as seen in the ANOVA table (Table S5). As detailed in the Pareto chart (Fig. 2a), the extraction yield was significantly influenced by all extraction parameters. When looking at the response surface in Fig. 2a, the following trend is clear: a lower influence of T at lower fractions of E, and a greater influence of T towards pure ethanol, leading to higher extraction yield.

The solvent choice is a key step for a successful extraction, and in this sense, the rule of the thumb is "like dissolves like", i.e., solvents with high dielectric constant (polarity) for polar analytes, and vice versa (Mustafa & Turner, 2011). Some factors can further improve this process, as observed with temperature and pressure.

Temperature affects the mass transfer properties by modifying some solvent properties, such as surface tension, diffusivity, and viscosity. That is, while surface tension and viscosity decrease, diffusivity increases by increasing the solvent temperature. All these changes promotes a faster mass transfer and improve wetting of the sample (Alvarez-Rivera et al., 2020). The use of high temperatures also improves the extraction efficiency as it helps the disruption of the analyte-sample matrix interactions caused by van der Waals forces, hydrogen bonding and dipole attraction (Mustafa & Turner, 2011). The pressure was also crucial once this parameter in PLE is not only responsible to keep the solvent in the liquid state above its boiling point but also contributes to enhance the extraction yield by controlling the formation of air bubbles that hinder the solvent from reaching the analyte (Mustafa & Turner, 2011).

The results of yield in PLE suggests that most of the compounds from tomato pomace (desired or not) have higher solubility in solvents with higher dielectric constant such as ethanol, and this solubility has considerably improved as a result of the high temperature and pressure.

3.2.2.2. Antioxidant activity. Tables S6, S7, and S8 show that all the polynomial models fitted to the experimental data of DPPH, FRAP, and β-Carotene bleaching method, thus adequately describing the influence of the factors on the responses.

For the DPPH method, the Pareto chart (Fig. 2b) illustrates that all the extraction parameters were significant. The response surface shows that the influence of T was positive, i.e., the higher the T, the higher the antioxidant activity. In contrast, the lower flow rates the higher the antioxidant activity, as can be seen by the negative influence of the factor F. Such effect may be due to an extension in the contact time between the solvent and the sample, which allowed the extraction of antioxidant compounds intrinsically linked to the plant matrix.

Although the use of solvents at high temperatures might decrease the extraction selectivity and affect the thermo-labile compounds (Mustafa & Turner, 2011), such effects were not observed in the antioxidant activity by the FRAP method, which can be seen by the response surface (Fig. 2c). The T and T^2 factors were not only the most significant extraction factors, as demonstrated by the Pareto chart (Fig. 2c), but their influence was positive, that is, higher temperatures favored the obtainment of reducing compounds of the ferric ion, a potent metal ion capable of initiating lipid oxidation.

The Pareto chart in Fig. 2d reveals that the results of the β-carotene method were affected by all extraction parameters in the following descending order: F^2, E, and T.

The response surface (Fig. 2d) illustrates the relevance of the flow rate, where the surface concavity facing upwards indicates that the lower and mainly the higher flows favored the response.

The influence of E was linear and negative, which represents an opposite behavior to those observed for the other antioxidant methods. In addition, this behavior also goes in the opposite direction of extraction yield. Nevertheless, our results are in good agreement with Pandya's (2017) findings, confirming that although the pure ethanol has favored the extraction in quantitative terms, the solubility of the antioxidant compounds and their diffusivity tended to be higher in ethyl acetate.

3.4. Determination of the global optimum

Based on the regression model, a multiple-response optimization was carried out using the desirability function. In this context, the maximal conditions for MAE were T = 90 °C; E = 90:10 v/v; and t = 5 min. With a desirability value of 0.93, the selected RSM model may be accurately applied for MAE extracts with maximum yield and antioxidant activity.

The predicted responses were 6.12 % for yield, 15.45 μmol/g for DPPH, 23.73 μmol/g for FRAP, and 41.07% for β-carotene, which are quite similar to the values obtained from assay 4 of the experimental design (Table 3). Despite the optimum conditions were at the experimental region limit, the experimental values from assay 4 are within the confidence interval of all predicted responses. Therefore, new extractions under the optimum conditions were not checked experimentally.

The maximal conditions for PLE were T = 90 °C, E = 44.7 v/v, and V = 2 mL/min^{-1}, with a high desirability value of 0.85. The predicted responses (Table 3) were 14.35 %, 17.42 μmol/g, 22.55 μmol/g, and 46.49%, for yield, DPPH, FRAP, and β-carotene, respectively. The experimental values from assay 6 are very similar to the predicted responses, and they are within the interval confidence, as for the optimization of MAE. Therefore, considering that assay 6 conditions already encompass the conditions that maximize yield and antioxidant activity, a new extraction was not performed under the conditions predicted by the model.

3.5. Analysis of extracts obtained under optimum conditions

3.5.1. Yield and antioxidant activity

In general, the oven-dried samples demonstrated higher extraction yield values, but lower antioxidant activity, as observed in Table 4. The longer drying period in which the oven-dried sample was subjected (12

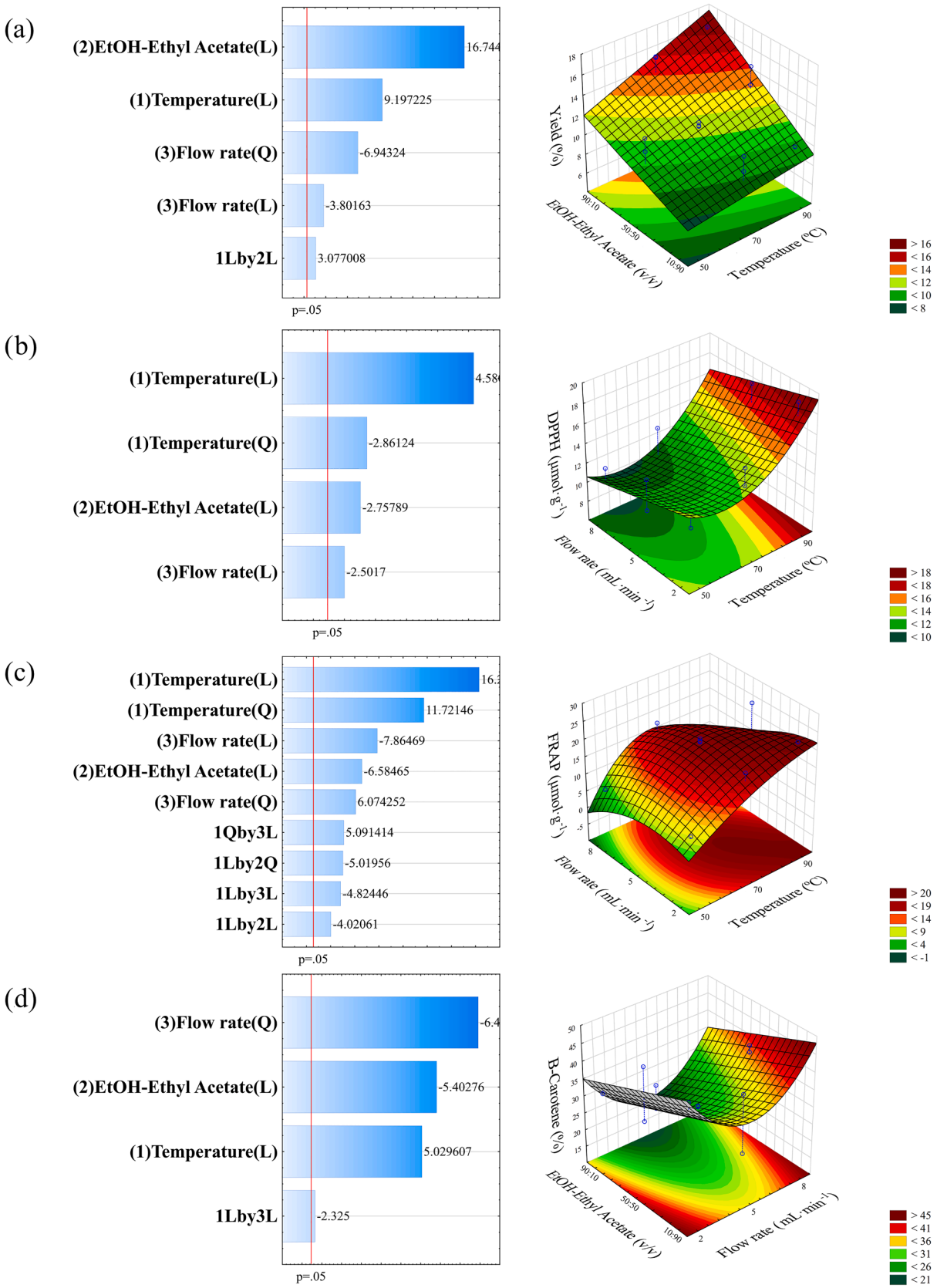

Fig. 2. Pareto charts for the response variables yield (2a), DPPH (2b), FRAP (2c), and β-Carotene (2d) studied in BBD of PLE, and their correspondent response surfaces.

Table 3
Values predicted by the desirability function (global optimum) and observed in assay 4 of the microwave-assisted extraction (MAE) and in assay 6 of the Pressurized liquid extraction (PLE).

Response		R^2	R^2 adjusted	Predicted values	Confidence Interval (CI)	Observed values
MAE[1]	Yield (%)	0.92	0.89	6.12	5.70–6.53	6.42
	DPPH (μmol/g)	0.97	0.96	15.45	14.42–16.48	15.06
	FRAP (μmol/g)	0.88	0.83	23.73	21.47–25.99	24.52
	β-Carotene (%)	0.89	0.85	41.07	37.89–44.24	39.82
PLE[2]	Yield (%)	0.93	0.90	14.35	13.09–15.62	15.18
	DPPH (μmol/g)	0.63	0.49	17.42	14.18–20.67	19.10
	FRAP (μmol/g)	0.57	0.40	22.55	14.24–30.85	22.01
	β-Carotene (%)	0.91	0.88	46.49	41.93–51.06	46.51

DPPH: 2,2–Diphenyl-1-picrylhydrazil; FRAP: ferric reducing antioxidant power; β-Carotene: beta carotene bleaching method.
[1] MAE assay 4: T (temperature) = 90 °C, E (solvent mixture ethanol:ethyl acetate) = 90:10 (v/v), t (extraction time) = 3 min.
[2] PLE assay 6: T (temperature) = 90 °C, E (solvent mixture ethanol:ethyl acetate) = 50:50 (v/v), F (solvent flow rate) = 2 mL/min.

Table 4
Influence of drying methods on yield, antioxidant activity methods (DPPH, FRAP, β-Carotene), and Lycopene content of the Soxhlet extracts and the global optimum extracts from microwave assisted extraction (MAE) and pressurized liquid extraction (PLE).

Drying method	Extraction technique	Yield (%)	DPPH (μmol TE/g)	FRAP (μmol TE/g)	β-Carotene (%)	Lycopene ($\mu g \cdot g^{-1}$)
Spouted bed	MAE[1]	6.42 ± 0.25^{e}	15.06 ± 0.48^{d}	24.52 ± 0.56^{e}	39.82 ± 0.35^{b}	59.66 ± 0.42^{a}
	PLE[2]	15.18 ± 0.22^{d}	19.10 ± 0.23^{b}	22.01 ± 0.72^{f}	46.51 ± 0.70^{a}	20.09 ± 0.88^{c}
	Soxhlet (EtOH)	21.01 ± 0.03^{b}	16.37 ± 0.28^{c}	30.03 ± 0.53^{c}	23.80 ± 0.35^{f}	10.75 ± 0.77^{e}
	Soxhlet (EA)	15.72 ± 0.02^{d}	18.43 ± 0.29^{b}	43.37 ± 0.18^{a}	33.46 ± 0.23^{d}	14.88 ± 0.75^{d}
Oven	MAE[1]	7.15 ± 0.55^{e}	12.04 ± 0.33^{e}	12.02 ± 0.40^{h}	29.79 ± 0.33^{e}	26.03 ± 0.54^{b}
	PLE[2]	16.91 ± 0.06^{c}	21.44 ± 0.42^{a}	26.55 ± 0.56^{d}	36.44 ± 0.47^{c}	7.30 ± 0.81^{f}
	Soxhlet (EtOH)	23.68 ± 0.39^{a}	11.18 ± 0.36^{f}	17.97 ± 0.50^{g}	20.68 ± 0.66^{g}	5.66 ± 0.52^{g}
	Soxhlet (EA)	20.66 ± 0.35^{b}	16.75 ± 0.49^{c}	37.71 ± 0.76^{b}	29.05 ± 0.58^{e}	$6.79 \pm 0.15^{f,g}$

DPPH: 2,2–Diphenyl-1-picrylhydrazil; FRAP: ferric reducing antioxidant power; β-Carotene = beta carotene bleaching method; EtOH = pure ethanol; EA = pure ethyl acetate.
The results represent the average of triplicate assays ± standard deviation.
Values with equal letters in the same column do not differ significantly from each other ($p < 0.05$).
[1] MAE assay 4: T (temperature) = 90 °C, E (solvent mixture ethanol:ethyl acetate) = 90:10 (v/v), t (extraction time) = 3 min; [2]PLE assay 6: T (temperature) = 90 °C, E (solvent mixture ethanol:ethyl acetate) = 50:50 (v/v), F (solvent flow rate) = 2 mL/min.

h vs. 1.5 h of the spouted bed-dried sample) may have led to greater degradation of bioactive compounds. The spouted bed dryer generates high heat and mass transfer rates throughout the process, allowing the use of higher temperatures but for a short period, which ends up reducing the possibility of a marked degradation of thermo-labile compounds, as in conventional drying methods (Mujumdar, 2006). Therefore, the spouted bed drying technique is the a suitable option for drying tomato pomace.

The Soxhlet results indicate that the use of ethanol led to a greater amount of compounds extracted from the matrix, and consequently, a higher yield. On the other hand, the extracts obtained with ethyl acetate showed significantly higher antioxidant activity levels. This may be due to the ability of ethyl acetate on extracting classes of compounds with different polarities and remarkable antioxidant activity, such as terpenoids and flavonoids (Widyawati et al., 2014).

Table 4 reveals that the antioxidant activity values were statistically different, demonstrating that each extraction technique provided products with distinct constitutions, and in this case, the PLE extracts stood out with the most prominent antioxidant activity.

In PLE, the main role of high pressure is to keep the solvent in the liquid state above its boiling temperature, however, it also presses the solvent toward hardly accessible regions inside the matrix solubilizing the analytes that would not be easily extracted at ambient pressure (Santos et al., 2021). Furthermore, the high pressure controls the formation of air bubbles within the matrix, which reduces the extraction efficiency by preventing the solvent from reaching the analyte (Santos et al., 2021).

3.5.2. Lycopene quantification

3.5.2.1. Influence of drying techniques. In this study, the spouted bed-dried extract obtained with acetone had 89.14 ± 0.56 μg lycopene/g of extract, thus making the reference lycopene value.

As can be noted in Table 4, the lycopene content of the extracts obtained from oven-dried samples was significantly lower than those obtained from the spouted bed-dried samples. Although the conventional technique had been performed at a lower temperature, these results suggest that its long drying time (12 h vs. 1.5 h for the non-conventional technique) was essential in the loss of lycopene from the raw material.

3.5.2.2. MAE and PLE vs. Conventional extraction technique. Table 4 shows that the lycopene content in MAE and PLE extracts was significantly higher than those obtained by Soxhlet. Once the conventional technique was carried out at the solvent boiling point, i. e. below the temperature used by the non-conventional techniques, it suggests a possible lycopene degradation in Soxhlet extracts due to their much longer extraction time. These results are in agreement with Madia et al (2021), which reported that a long time and high temperature applied during Soxhlet extraction can degrade heat-sensitive compounds. According to Papaioannou et al (2016), one of the disadvantages of using conventional lycopene extraction techniques is its long process time, which ends up making it difficult to recover this labile compound. In this way, non-conventional extraction techniques such as MAE and PLE become a viable alternative, since both can provide even higher lycopene recovery in a much shorter time.

Taking into account the relevance of the solvents, it can be seen a higher lycopene content in ethyl acetate extracts, which corroborates previous findings by Pandya (2017). In addition, this behavior is also in line with the antioxidant activity results, as discussed in section 3.5.1.

Interestingly, despite having been obtained with a higher fraction of ethyl acetate and having presented higher antioxidant activities, the

lycopene content of the PLE extracts was significantly lower than those obtained by MAE, indicating a possible degradation of lycopene in 50 min of extraction at 90 °C.

The MAE extract from the spouted bed-dried sample had the highest lycopene content, 59.66 μg lycopene/g of extract, which represents a recovery rate of 66.93%, in just 3 min of extraction, in comparison with the reference lycopene value previously mentioned (89.14 μg lycopene/g of extract), demonstrating the effectiveness of time and the viability of MAE in terms of lycopene recovery.

Scaglia et al (2020) reported a high lycopene recovery rate reaching 93%, using supercritical fluid extraction (SFE), one of the most used non-conventional extraction techniques to obtain lycopene from tomatoes. Despite being an environmentally friendly technique, SFE demands a high initial investment (Mustafa & Turner, 2011), which limits its use. Amiri-Rigi & Abbasi (2019) achieved 88% of lycopene from tomato pomace extract obtained under shaking (35 °C/30 min), and using olive oil microemulsion as a solvent; Lianfu & Zelong (2008) attained 89.4 to 97.4% of lycopene using ultrasound and microwave-assisted extraction (UMAE) techniques with ethyl acetate as a solvent; Naviglio et al (2008) reported rates from 8.5 to 19.5% using the pressurized water technique; From 65.2 to 75.8% of lycopene were recovered by Poojary & Passamonti (2015) using extraction with organic solvents. As can be seen, our results are in good agreement with previous findings in the literature.

According to Vinatoru et al (2017), MAE generates rapid and uniform heating, providing higher yields than conventional extraction techniques, in a shorter time, with less solvent, and with low energy consumption. Although the lycopene fractions obtained using MAE are similar to those obtained by several authors who used conventional extraction methods, the microwave technique stands out due to its process time being substantially shorter. For example, if a purification step were performed with the MAE extract, 16.76 g of extract (261.1 g of dried tomato pomace) would be needed to yield 1 mg of pure lycopene. The same purified lycopene is sold at approximately USD 142.0 (Sigma-Aldrich, 2021), demonstrating the feasibility of studying different drying and extraction processes of bioactive compounds from tomato pomace. In this sense, the present study offers a viable alternative for the process of extracting lycopene in a safe, more economical, and more sustainable way.

4. Conclusions

The objective of this study was duly achieved since the optimization of the PLE and MAE parameters allowed the obtainment of tomato pomace extracts with high antioxidant activity and enriched with lycopene.

First, the spouted bed drying demonstrated that this non-conventional technique was significantly more efficient than the conventional oven drying both in preserving the antioxidant compounds and the lycopene.

Through the RSM it was figured out that the parameters temperature and solvent were preponderant in the MAE responses and ended up showing a certain standard behavior. On the other hand, the PLE samples were significantly influenced by the three extraction parameters and did not show a clearly defined behavior for yield and antioxidant activity.

Next, it could be seen that the optimum MAE and PLE extracts stood out from Soxhlet extracts in quantitative and qualitative terms. In this sense, PLE extract showed the highest antioxidant activity content, while the MAE extract showed the highest lycopene content. Moreover, the MAE extract provided 59.66 μg lycopene/g, which represents around a 67 % recovery rate in 3 min of extraction.

Finally, the PLE and MAE extracts showed a high potential for application in the food industry as additives, as well as in the pharmaceutical industry. The promising results demonstrate that both the prior drying technique and the extraction techniques were successful in adding value to tomato pomace, a rich source of bioactive compounds but often discarded.

Declaration of Competing Interest

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

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2022.100237.

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