Revista Mexicana de Ingeniería Química

OPTIMIZATION OF β-CAROTENE FROM 'ATAULFO' MANGO (*Mangifera indica* **L.**) BY-PRODUCTS USING ULTRASOUND-ASSISTED EXTRACTION

OPTIMIZACIÓN DE β-CAROTENO DE LOS SUBPRODUCTOS DE MANGO (*Mangifera indica* **L.**) 'ATAULFO' UTILIZANDO LA EXTRACCIÓN ASISTIDA POR ULTRASONIDO

G. Mercado-Mercado[1], E. Montalvo-González[1], J.A. Sánchez-Burgos[1], R.M. Velázquez-Estrada[1], E. Álvarez-Parrilla[2], G.A. González-Aguilar[3], S.G. Sáyago-Ayerdi[1]*

[1]*Tecnológico Nacional de México/Instituto Tecnológico de Tepic, Av. Tecnológico 2595, CP 63175, Tepic, Nayarit, México.*
[2]*Departamento de Ciencias Químico Biológicas, Instituto de Ciencias Biomédicas, Universidad Autónoma de Ciudad Juárez, Anillo envolvente PRONAF y Estocolmo, Ciudad Juárez, Chih 32310, México.*
[3]*Coordinación de Alimentos de Origen Vegetal, Centro de Investigación en Alimentación y Desarrollo A.C., Carretera a La Victoria Km 0.6 CP 83304, Hermosillo, Sonora, México.*

Abstract

The present study investigated β-carotene content of 'Ataulfo' mango by-products, obtained from a local industry. β-carotene was evaluated by HPLC-PDA. Extraction of β-carotene from mango peel and paste was optimized using ultrasound-assisted extraction (UAE). The influence of three independent variables was evaluated: extraction time (min, X_{ET}), sonication amplitude (%, X_{SA}), and pulse cycle (X_{PC}). The β-carotene extraction was optimized using a response surface methodology. The optimal conditions to achieve the maximal content of β-carotene were X_{ET} (30 min), X_{SA} (30%), X_{PC} (0.8) for both byproducts. Experimental results (peel 19.13 ± 0.41 mg/g dry weight (DW) and paste 6.60 ± 1.60 mg/g DW) correlated positively (R=0.99) with predicted values. The extraction efficiency of β-carotene was significantly higher in UAE than conventional solvent extraction. These results indicate that UAE of β-carotene from mango peel and paste can be used to improve the yield in the extraction of carotenoids in mango by-products.

Keywords: Ataulfo, ultrasound-assisted extraction, β-carotene, by-products, mango.

Resumen

En el presente estudio se investigó el contenido de β-caroteno de los subproductos de mango 'Ataulfo', obtenidos de una industria de la localidad. El β-caroteno fue evaluado por HPLC-PDA. La extracción de β-caroteno de la cáscara y pasta del mango fue optimizado utilizando la extracción asistida por ultrasonido (UAE). Se evaluó la influencia de tres variables independientes: el tiempo de extracción (min, X_{ET}), amplitud de sonicación (%, X_{SA}) y el ciclo de pulso (X_{PC}). La extracción de β-caroteno fue optimizado usando la metodología de superficie de respuesta. Las condiciones óptimas para alcanzar el contenido máximo de β-caroteno fueron X_{ET} (30 min), X_{SA} (30%), X_{PC} (0.8) para ambos sub-productos. Los resultados experimentales (cáscara 19.13 ± 0.41 mg/g peso seco (DW) y la pasta 6.60 ± 1.60 mg/g DW) se correlacionaron positivamente (R=0.99) con los valores previstos. La eficiencia de extracción de β-caroteno fue significativamente mayor en UAE que en la extracción convencional por solventes. Estos resultados indican que el UAE de β-caroteno de la cáscara y subproductos de la pasta del mango pueden ser usados para mejorar el rendimiento en la extracción de carotenoides en los sub-productos del mango.

Palabras clave: mango, 'Ataulfo', extracción asistida por ultrasonido, β-caroteno, subproductos, *Mangifera indica*.

1 Introduction

'Ataulfo' mango (*Mangifera indica* L.) is a tropical fruit highly demanded by consumers for its good sensorial and nutritional properties. Commonly, the juice is obtained as main product during its industrial processing, and by-products (BP) as peel, kernel, and paste are generally discarded (Jahurul *et al*., 2015). It has been reported that these by-products (peel and

paste) are a good source of bioactive compounds such as polyphenols and β-carotene (βC) (Jahurul *et al*., 2015; Moo-Huchin *et al*., 2015). The paste is considered as the fibrous residue, after removing the mango pulp in the fruit. The biological importance of βC is that is a vitamin A precursor, and additionally has different biochemical functions, such as, inhibition of lipoxygenases, stimulation of connexin to increase gap junction formation, antioxidant properties, among others (Chedea *et al*., 2013; Hewavitharana *et al*., 2013).

Mango by-products (BP) have been proposed as an alternative to be used as ingredients in the formulation of new food products to fortify flours and/or bakery products, due to their high dietary fiber (DF) and bioactive compounds (carotenoids and phenolic compounds) (Ramírez-Maganda *et al*., 2015) content. According to references, the consumption of mango peel could provide sixfold more DF than bioactive compounds (Ramírez-Maganda *et al*., 2015). Blancas-Benítez *et al*. (2015) showed an increase in DF content (13.39%) and phenolic compound content (626 mg/100 g) by adding a proportion of pulp and mango peel and paste powders (25:75 w/w) in snacks. Also, other study observed similar results in the addition of mango peel in muffins (Ramírez-Maganda *et al*., 2015). On the other hand, Mercado-Mercado *et al*. (2018) determined a high carotenoid content in peel and paste during an in vitro digestion model. However, DF is a critical component in the release of carotenoids, and the extraction method had been important to improve their release (Morales-de la Peña *et al*., 2018).

The conventional solvent extraction (CSE) consists in a simple maceration or heating and is commonly used to extract βC from different sources. However, the extraction conditions such as: time, solvents, methods, and food matrix, are factors that influence the removal of this compound (Kumar *et al*., 2017). Moreover, the βC content in mango peel depends on the variety/cultivar and maturity stage. It has been reported the βC content from 13.09 mg/100 g in 'Bambangan' mango peel to 0.953 mg/100 g in 'Rosa' mango peel, both varieties from Brazil (Ribeiro da Silva *et al*. 2014). The βC content reported for 'Ataulfo' mango pulp was 4.2 mg/g dry weight (DW) (Ornelas-Paz *et al*., 2008). Previous studies have reported the βC content of 'Ataulfo' pulp and their individual contribution to the antioxidant capacity and health benefits (Ornelas-Paz *et al*., 2008; Palafox-Carlos *et al*., 2012).

On the other hand, the use of ultrasound-assisted extraction (UAE) is an emerging technology that has extensively been used in the last ten years. Cavitation of the UAE is a dynamic phenomenon of formation, growth and collapse of bubbles in a liquid with a high concentration of energy within a fluid (Sánchez-Madrigal *et al*., 2017). The generation and development of cavitation are functions of the liquid properties, the presence of pollutants, ambient pressure and temperature, among others. Different carotenoids from different matrices have been obtained using UAE (Lopes *et al*., 2014; Zhao *et al*., 2006). However, the effect of UAE on carotenoids extraction of mango by-products, have been scarcely studied. The advantage of UAE is that cavitation, breaks the cell membrane favoring the extraction of βC with a considerably reduction of time, use of solvents, and increasing the content of interest compounds (Krasulya *et al*., 2016). The use of UAE in plant-foods has demonstrated that also offers a high reproducibility, extraction of heat labile components, significant savings in energy and the maintenance of the system and, is considered as a green process (Deng *et al*., 2015). Thus, optimization of the extraction parameters represents a critical step in the development of the method for a specific sample. Response surface methodology (RSM) can optimize the complex extraction procedures by investigating the variables and their interactions simultaneously during the extraction of bioactive compounds from plant tissues (Khuri, 2017). Hence, the aim of this work was to evaluate the effect of UAE to increase the extraction of βC from mango BP (peel and paste), using RSM to optimize the extraction parameters.

2 Materials and methods

2.1 Raw materials

'Ataulfo' Mango BP (peels and paste) were obtained from a local industry (MexiFrutas S.A. de C.V., Nayarit, Mexico) and transported to the Laboratory in the Technological Institute of Tepic. Peel and paste were cleaned, and dried in a convection oven (WTR910, Scorpion Scientific, A-52055, USA) at 60°C, 15 h for peel and 7 h for paste, conditions previously established. Moisture content of peel and paste dried was 3.5% and 2.4%, respectively. Dried samples were sieved through a 500 μm mesh, stored in a hermetic container, and frozen at -20°C until further

analysis.

2.2 Mango BP conventional solvent extraction of carotenoids

Carotenoids were carried out extracting and quantifying according to De Ancos *et al.* (2000) methodology with some modifications. Each BP (500 mg) was mixed with tetrahydrofuran (THF) (1:10 sample-solvent ratio) and butylated hydroxytoluene (BHT, 0.1 mg/g of sample) during 30 min. After the time, the mixed were centrifuged (Hermle®, PM18012, Germany) at 4000 rpm for 15 min at 5 °C, and the extracts were transferred into red flasks at room temperature. The residue was re-extracted until colorless, and concentrated using a rotary evaporator (Buchi, R-124 Flawil, Switzerland) at 35°C. The concentrated samples were re-dissolved in diethyl ether analytical grade (2 mL) washed with sodium chloride (10%, w/v), and dried with anhydrous sodium sulfate (500 mg). The solvent was evaporated using a rotary evaporator and the residue was re-dissolved with acetonitrile and was filtered through a 0.45 μm membrane (Whatman International Limited, Kent, England) using a vacuum pump (WP6211560, Millipore, USA) and analyzed immediately by HPLC-PDA as follows.

2.3 Identification and quantification of βC by HPLC-PDA

βC was identified and quantified in an HPLC system (Dionex®, ICS-5000, USA) with photodiode array detector (PDA) (Dionex®, Ultimate 3000, USA), and data were integrated in the Chromeleon Management software (Shimadzu LD Driver for Chromeleon^{TM} v6.8, USA, 2006). Separations peaks were performed on a C30 column (5 μm particle size Vydac 201TP54, 250 mm in length × 3.0 mm in diameter, Thermo Scientific®, USA) with a C30 guard column (20 × 4.6 mm; Thermo Scientific®, USA), at 25°C. The mobile phase consisted of two eluents: acetonitrile (100%, A) and methanol:ethyl acetate (50:50 % v/v, B) in a gradient elution and the temperature of the column was kept at 25 °C. The gradient elution used with this column was 85% A, 15% B at 7 min, followed by linear gradient to 65% A, 35% B to 15 min, at 10 min the gradient changed to 25% A, 75% B and was maintained for 50 min, at 50 min the gradient was changing to 25% A, 75% B followed by a linear gradient to 0% A, 100% B for 5 min and return to initial conditions for 10 min. A re-equilibration (5

min) was carried out at initial concentrations of 85% A, 15% B. The column temperature was maintained at 25°C, running time was 70 min in a flow rate of 0.8 mL/min, and injection volume was 10 μL. βC was identified by comparing the retention times of sample extraction solution to these of βC standard in the wavelength at 450 nm, obtaining the common maximum absorbance. The mobile phase, extracts, and βC standard were filtered through a syringe sterile filter with a 0.45 μm pore size (Millipore Indústria e Comércio Ltda®, San Páulo, Brazil). The concentration range for the βC standard curve was 0.0062-0.12 mg/mL. The βC content was expressed as mg/g dry weight (DW). βC peaks were identified by their absorption spectrum.

2.4 Ultrasound-assisted extraction (UAE) of carotenoids

UAE was adapted using a previously described methodology (De Ancos *et al*., 2000). The UAE were carried out using an ultrasonic processor UP 400S (Hielscher GmbH, Teltow, Germany) composed with a sonotrode of a diameter from 7 mm. The maximum power and frequency of the ultrasonic processor was 400 W and 24 kHz with adjustable amplitude from 20 % to 100 %. Briefly, 500 mg of BP (peel or paste) was mixed with THF (1:10 sample-solvent ratio) and BHT (0.1 mg/g of sample) and the ultrasonic transducer (H7 Tip 7, Hielscher, Teltow, Germany) was immersed 20 mm into the bottom of a beaker, using maximum amplitude of 175 mm and acoustic power density of 300 W/cm^2. The sample was kept in constant stirring. The temperature was controlled by a digital thermoregulator (B401L, Firstek, Taiwan) at 5°C ± 3, connected to the container where an iced water bath was located to ensure constant temperature, at dark to avoid potential light-induced damages of the extract. The temperature was monitored every 5 min intervals for each UAE treatment. The extract obtained was filtered (Whatman No. 1HP7 9NA, UK) under vacuum (F1-125 Faga-Lab, Mocorito Sinaloa, Mexico) using a vacuum filtration unit (WP6211560, Millipore, Darmstadt, Germany). The supernatant was concentrated at 35°C to remove the solvents using a rotary evaporator (Buchi, R-124 Flawil, Switzerland). The sample was freeze-dried (FreeZone 6, Labconco, Fort Scott, Kansas, USA), ground, and stored at -22°C until analysis. All the extractions were carried out in triplicate and they were used for further carotenoid analysis as mentioned above.

Table 1. β-carotene content (βC) extracted from mango peel and paste with CSE and UAE under optimum conditions (30 min, 30 %, 0.8).

Analysis	CSE		UAE	
	Peel	Paste	Peel	Paste
βC content (mg/g DW)	1.21 ± 0.12Ba	0.56 ± 0.007Bb	19.13 ± 0.41Aa	6.60 ± 1.60Ab

*Data is expressed as mean ± SD (n = 3). Uppercase letters indicate significant difference between treatments. Lowercase letters indicate significant difference between samples in the same treatment (p < 0.05); CSE: Conventional solvent extraction; UAE: Ultrasound-assisted extraction.

2.4.1 Experimental design

A 3^{3-1} fractional factorial design (defining factor AB^2C^2) was used to determine the optimal UAE conditions with three levels (-1, 0, +1). Previous data suggested to determine the independent variables and preliminary experiments were performed to acquire the optimum ranges of three single variables: the extraction time (X_{ET}, 10, 20, 30 min), sonication amplitude (X_{SA}, 30, 65, 100%), and pulse cycle (X_{PC}, 0.4, 0.6, 0.8 s) (Table 1). Based on the manufacturer's effective power rating, the ultrasonic power for the three power settings inside the extract containers were 18 W/cm^2, 39 W/cm^2, and 48 W/cm^2, respectively. βC (mg β-carotene/g DW) was selected as the response for the combination of the independent variables. A response surface methodology (RSM) was applied to determine the optimal conditions, and a second order polynomial (Eq. 1), which includes all the terms, was used to predict the response:

$$Y = \beta_0 + \sum_{i+A}^{B} \beta_i X_i + \sum_{i=A}^{B} * \sum_{i=A \neq i}^{B} \beta_{ij} X_i + E \qquad (1)$$

Where Y is the predicted response for βC content, β_o is a constant term, X_i is coded values for the factors (X_{ET}, X_{SA}, and X_{PC}), β_i is a main effect for the evaluated variable, and β_{ii} and β_{ij} are the interaction effects coefficients. Model adequacy was estimated using F ratio. Lack of fit test was used to determine significant interactions in the model and coefficient of determination (R-square and R-Adjust) represented at 5% level of significance.

2.5 Statistical analysis

The data were expressed as mean ± standard deviation (n = 4). One-way ANOVA and Fisher's least significant difference (LSD) method were used to examine the differences between samples and groups (p<0.05). All statistical analyses were done with Statistic 10.0 (Stat Soft. Inc., Tulsa, OK, USA).

3 Results and discussion

3.1 Conventional solvent extraction of βC

THF was selected as the most suitable solvent for the optimization of extraction due to its highest βC extraction efficiency compared with other investigated solvents (Winggvist, 2011). BHT was used to prevent the oxidation of fatty-compounds and consequently, cause a reaction that would lead to the formation of isomers or loss of β-carotene (Delgado *et al.*, 2015; Vasantha-Rupasinghe and Yasmin, 2010). Table 1 shows of βC from mango peel and paste, which was used for comparison in all the analyses. βC content was 1.29 ± 0.12 mg/g DW and 0.56 ± 0.007 mg/g DW for peel and paste, respectively. In contrast, Singh *et al.* (2013) observed that during the CSE the βC content decreases 1.5 times after the use of conventional drying. βC content in peel was two-fold higher than in paste; this difference could be attributed to the fact that in the peel are chromoplasts where carotenoids can protect their structure (Guiamba & Svanberg, 2016). Thus, the drying process was also an important factor that favored the extraction of carotenoids from mango BP, since there are no matrix components (i,e, water, water-soluble compounds) that interfere with THF to extract these compounds (Yan *et al.*, 2016). Drying also reduces extraction times leading to increased mass transfer during extraction processes due to changes in the diffusivity coefficient of cell membranes, increased cell membranes permeability on THF (de Oliviera *et al.*, 2016). The lower βC content found in mango paste can be explained considering that most of βC was extracted from the pulp during the industrial processing of mango (Ornelas-Paz *et al.*, 2008). Mango paste has a yellow color given by the presence of carotenoids, however; CSE required prolonged time to complete the extraction of the βC, because dietary fiber apparently can difficult the diffusion of the THF throughout the tissue and in consequence the

Table 2. Effect of process variables of ultrasound-assisted extraction on β-carotene (βC) content in mango 'Ataulfo' peel and paste.

No.	Ultrasound conditions			Ultrasonic power (W/cm^2)	Peel	Paste
	X_{ET}^1	X_{SA}^2	X_{PC}^3		βC content (mg/g DW)	
1	10	30	0.4	18	6.28 ± 0.39a	0.19 ± 0.05b
2	10	65	0.6	39	4.06 ± 0.88a	1.78 ± 0.16b
3	10	100	0.8	48	4.61 ± 0.41a	1.98 ± 0.41b
4	20	30	0.4	18	1.54 ± 0.23b	5.60 ± 0.83a
5	20	65	0.6	39	1.21 ± 0.15b	4.47 ± 0.43a
6	20	100	0.8	48	6.18 ± 0.65a	4.48 ± 0.38b
7	30	30	0.4	18	19.86 ± 0.3a	7.12 ± 0.12b
8	30	65	0.6	39	13.26 ± 0.9a	1.51 ± 0.18b
9	30	100	0.8	48	8.86 ± 0.86a	1.65 ± 0.14b

1XET: exposition time (min); 2XSA: sonication amplitude (%); 3XPC: pulse cycle. Data expressed as mean ± SD (n=4); Lowercase letters indicate significant difference between samples in the same treatment ($p < 0.05$).

extraction of carotenoids (Sudha *et al*., 2015). However, THF favors of breaking of the intramolecular bonds of hydrogen and could transfer the compounds to the organic phase through the formation of hydrogen bonds between the oxygen atom of the THF and the hydrogen atom of carotenoids (Jiang *et al*., 2018).

3.2 Effect of UAE on βC content of 'Ataulfo' mango BP

Table 2 shows the effect of UAE variables, which were analyzed in a 3^{3-1} fractional factorial design. βC concentration in all treatments are ranged from 1.21 ± 0.15 mg/g DW (X_{ET} 20 min, X_{SA} 20%, X_{PC} 0.6) to 19.86 ± 0.29 mg/g DW (X_{ET} 30 min, X_{SA} 30%, X_{PC} 0.4) in peel. These values are the results of different mechanisms that can take place during the extraction: a) a greater amplitude, can generate heat increase in the medium, b) the same extraction time of other compounds such as organic acids and vitamin C can take place, c) both heat and presence of organic acids can cause carotenoid oxidation, and d) interaction between the extraction time, and high pulse cycle may attribute to the oxidation and degradation of carotenoids (Mohamad-Said *et al*., 2016; Abulizi *et al*., 2014). It has been observed a negative effect when UAE and temperature is combining, reducing the carotenoids content (Alim *et al*., 2016). Therefore, the reduction of the βC

content could be attributed to the oxidation mechanism mentioned above, limiting the effect of temperature. The aim of temperature control was to reduce Maillard and oxidation reactions of other components present in mango BP. It should be noted that BHT only favors stabilizing or inhibiting the oxidation of lipid compounds (fatty acids, carotenoids). Cavitation hydrolyzes the complex structures of polysaccharides when the temperature is not kept under control or when there is a sonothermal starting from the 40 °C (Sánchez-Madrigal *et al*., 2017). Likewise, the sonicate process is not a selective process but rather cavitation occurs throughout the matrix, which can generate a cascade of reactions that cause structural changes in the carotenoids or reduce their content. Therefore, it was decided to maintain a temperature where this phenomenon is avoided and ensure a good extraction of β-carotene. However, in our study we focused on the effect of the UAE variables maintaining the temperature constant (5°C ± 3) to prevent the oxidation of these compounds (Takeungwongtrakul & Benjakul, 2016). This research aims to report the advantage of UAE in the release of carotenoids. It is known that mango BP are rich in DF, which resists the digestion process, preventing carotenoids from being released and absorbed. Therefore, the use of UAE improves the release of carotenoids in the mango BP, doing highly bioaccessible and potentially bioavailable, so that they can exert biological functions (Mercado-Mercado *et al*., 2018).

Table 3. Predicted and experimental values at optimal conditions of UAE of β-carotene (βC) from mango 'Ataulfo' peel and paste.

	Response variable	X_{ET}	X_{SA}	X_{PC}	Predicted value (mg/g dry base)			Experimental value (mg/g DW)
						Lower	Upper	
Peel	βC	30	30	0.8	19.86	18.36	21.37	19.13 ± 0.41
Paste	βC	30	30	0.8	7.12	6.64	7.61	6.60 ± 1.60

[1]Mean ± SD (n=3).

Also, the combination of the UAE variables may release other compounds with antioxidant potential (Krasulya *et al*., 2016) and fluctuations of small X_{PC} can arrive quickly to the intra-thylakoid space of chloroplasts, facilitating the release of the βC present in the thylakoid membranes (Carail *et al*., 2015; Dey & Rathod, 2013).

The results in the experimental conditions showed that the highest βC content for paste was 7.12 ± 0.12 mg/g DW (X_{ET} 30 min, X_{SA} 30%, and X_{PC} 0.4) and the lowest was 0.19 ± 0.05 mg/g DW (X_{ET} 10 min, X_{SA} 30 %, and X_{PC} 0.4). For this reason, the difference in the βC content was influenced by the effect of UAE and this can be due attributed to the previous industrial process that suffered the paste. This can be explained by the effect of UAE produced in the solubility of βC and increase the permeability into the cell membrane improving the diffusion of the carotenoids (Azwanida, 2015; Dey & Rathod, 2013).

This process apparently accelerates the extraction of βC by low X_{SA}, and can correlate to the direct effect of UAE energy on βC, resulting in power dissipation through plant material matrix, which generate molecular movement and disrupts covalent and hydrogen bonds (Krasulya *et al*., 2016). Pasquet *et al* (2011) compared the extraction yield between UAE with low X_{SA} and the CSE process, and found higher yields with UAE for fucoxanthin of *Cylindrotheca closterium* and βC of *Dunaliella tertiolecta* with 45% and 50%, respectively. According to the results obtained, high X_{SA} (100%) had a negative effect on βC extraction in both BP. Some studies have shown that high sonication amplitudes increased the output of ultrasonic energy causing an increase of temperature, which could cause oxidation of some carotenoids (Chahine *et al*., 2016). βC content of paste obtained by UAE was lower than in peel, because during the mango processing the juice carried most of carotenoids of the food matrix (Bychkov *et al*., 2012). This could explain the lower extraction of

carotenoids during UAE in paste. Food matrix play an important role during extraction processes of bioactive compounds and the efficacy is directly correlated to the type of interactions and conjugations that take place with the macromolecules and composition of food matrix. The controlled conditions to apply UAE can improve the yield in the extraction of βC in mango BP as an alternative process to obtain this compound. ANOVA indicates that the variables (extraction time, sonication amplitude and pulse cycle) were influence to represent the relationship between the response values and the independent variables. The lack-of-fit values (p = 0.000) in both BP showed a high F-value and low p-value, indicating that the fitness of the model was highly significant. The high F-value (Peel 825.58, Paste 436.38) and low p-value (<0.0001 both BP) suggested that the regression model was highly significant in βC of BP. The high coefficient (R^2) and high-adjusted determination coefficient (R^2_{adj}) were 0.995 and 0.992, respectively to peel and 0.996 and 0.993, respectively to paste, indicating a high correlation between the predicted and experimental values.

3.3 Optimization of β-carotene extraction of mango BP by RSM

Fig. 1 shows the 3D surface plots, which allows visualizing the relationship between factors and βC content. It is apparent that the X_{ET} and X_{SA} showed strong positive influence on βC extraction. The response surface regression analysis was carried out to fit mathematical models to the experimental data with the aim of finding the optimal region for βC extraction. The following Eq (2) for peel and (3) for paste were obtained:

$$\beta C_{peel} = 19.65 - 3.21X_{ET} + 0.11X_{ET}^2 + 0.36X_{SA}$$
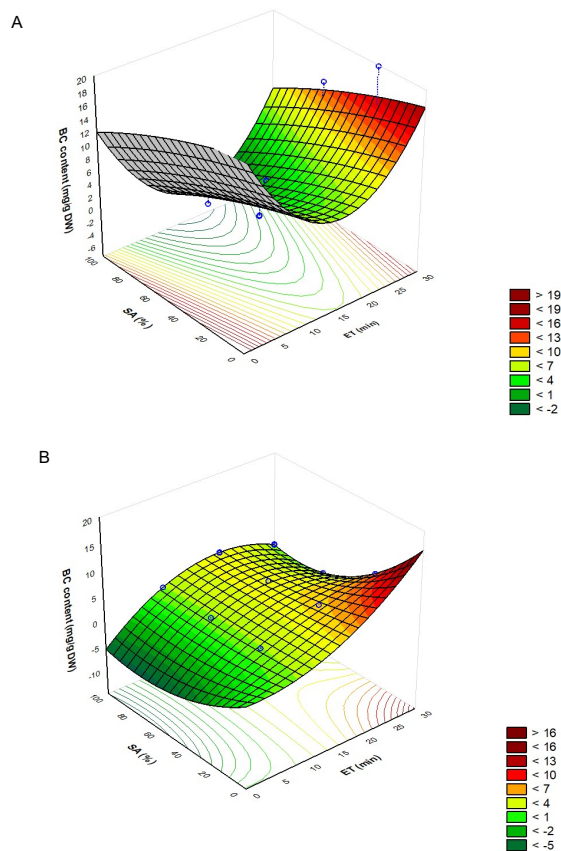$$- 0.003X_{SA}^2 - 0.01X_{ET}X_{SA} \quad (2)$$

Fig. 1. Surface plot as a function of extraction time (ET) and sonication amplitude (SA) of β-carotene content in mango 'Ataulfo' peel (A) and paste (B).

$$\beta C_{paste} = -12.74 + 1.08X_{ET} - 0.003X_{ET}^2 + 0.14X_{SA} + 0.002X_{ET}X_{SA} \quad (3)$$

Optimal process parameters were determined by simultaneously maximizing βC contents. Table 3 shows the experimental βC extraction in mango peel and paste. The predicted optimal conditions for simultaneous UAE were: X_{ET} (30 min), X_{SA} (30%), X_{PC} (0.8) for peel (19.13 mg/g DW of βC) and paste (6.60 mg/g DW of βC). The predicted and experimental values using the equation demonstrates that the model was successful capturing the correlation between parameters to the response and this was confirmed by the R^2 value of 0.99.

The statistically significant variables that influence the ultrasonic extraction process are time, temperature and power, respectively. Here, the application of pulsation during sonication does not produce any significant effects. The cavitation phenomena is responsible for enhancement in the extraction process, this can suggest that the supersonic jets from bubble implosions open spaces (capillaries) improving cell hydration. The statistical significance of the interactions between parameters reflects that enhancing the maximum content depends on various factors. The optimum yield was determined at the power of 100 W, the time of 30 min and the temperature of 15 °C which resulted the yield of about 21 mg/L.

3.4 Validation of predictive model

βC was extracted with the optimized parameters, where the feasibility of the experiment was taken into consideration. Table 3 shows the comparison between the predicted values of the RSM with the experimental values under these conditions. Mean values of 19.13 ± 0.41 and 6.60 ± 1.60 mg/g DW were obtained from experiments using peel and paste, respectively. The optimal predicted values and experimental data showed no significant difference (p > 0.05). Hence, RSM can be used to optimize βC extraction yield from Ataulfo' mango BP and minimize variability.

3.5 Comparison of CSE and UAE of βC in 'Ataulfo' mango BP

CSE was compared with the UAE under optimized conditions (Fig. 2). The extraction yield increased 91.51% and 93.72% in BPs, respectively, which demonstrates the advantages of using UAE to obtain higher amounts of βC. βC has been extracted with other emerging technologies, and it has been seen that the extraction yield was around 79% with supercritical CO_2 and 0.12% in pulsed electric fields (Mezzomo *et al.*, 2016; Durante *et al.*, 2014) these values were significantly lower with that obtained in this study. Therefore, the range of yield depends of the operational parameters (emerging technology, temperature, solvent, co-solvent, extraction time, others). Thus, UAE released the βC content in both BP because it can produce modifications in food matrices such as mechanical disruption of the cell membrane, reduce the size of the particles, providing better uniformity and stability, and allow the solubility or extraction of compounds (Carail *et al.*, 2015). Nafar *et al.* (2013) found that the importance of independent variables on the effect of UAE on bioactive compounds could be ranked in the following order: ultrasound frequency > temperature > exposure time.
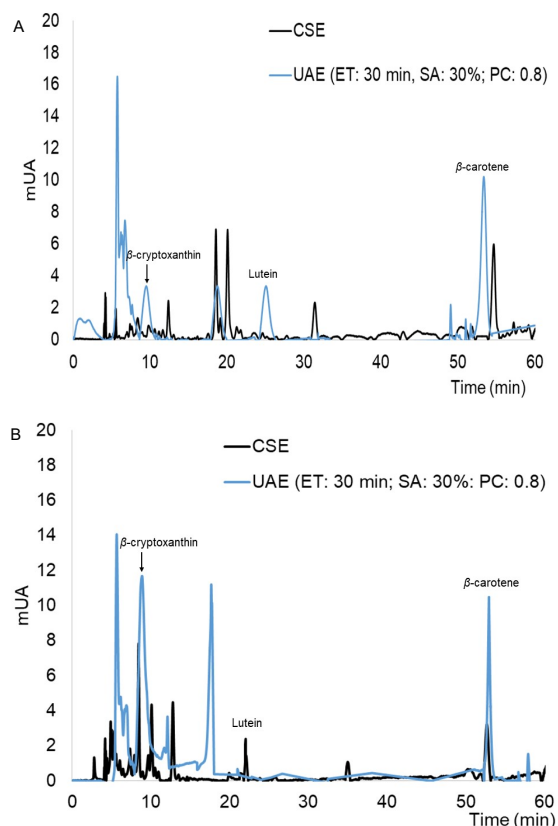
Fig. 2. Representative chromatogram of conventional solvent extraction (CSE) and UAE-extracted carotenoids under optimum UAE conditions from mango peel (A) and paste (B).

Fig. 2 shows the chromatogram comparing between CSE and optimal conditions of UAE of carotenoids in peel (A) and paste (B), respectively, where is noticeable that the βC and the carotenoids extraction were less efficient in the CSE and was remarkable in the samples with UAE. This can be explained because the effect that can exert the dietary fiber present in the samples can affect many physical properties including the viscosity, internal and external diffusivity, solubility and the surface tension of the carotenoids (Azwanida, 2015). Therefore, UAE is a biotechnological efficient technique to extract carotenoids and can be available to be used on an industrial scale (Michelon *et al.*, 2012). However, there are few studies that have implemented the UAE in micro-scale in the food industry, because it is difficult to standardize a condition to obtain a good yield of carotenoids from these BP because depends on UAE properties, process optimization, diameter of sonotrodes, viscosity, particle size distribution, surface tension, texture, composition of food matrix, and

kind and concentration of carotenoids (Vyas & Ting, 2018; Zavala-Lópes & García-Lara, 2017; Picó, 2013). Therefore, this study proposes only to analyzed the effect of the UAE conditions to observe the yield of βC of BP. Taking in consideration that 'Ataulfo' mango is one of the most important mango cultivar in México, and the amount of BP produced during the industrial processing, UAE appears to be a good alternative for the extraction of βC. Taking advantage of the high yield of βC can be used as an ingredient for the design of functional foods, and to help reduce the risk of pneumonia in smokers, reduce cancer risk, reducing the risk of developing, and others (Torregrosa-Crespo *et al.*, 2018).

Conclusions

In the present study, UAE was used to improve the β-carotene content of mango BP (peel and paste): a conventional method (using only solvents) and using UAE. The β-carotene content was 1.29 mg/g DW and 0.56 mg/g DW by peel and paste, respectively in conventional extraction. The RSM used for peel and paste optimized the experimental conditions using UAE to increase the β-carotene content. The optimal extraction conditions for β-carotene content for both BP were as follows: pulse cycle = 0.8, sonication amplitude = 30%, and extraction time = 30 min. Under these optimal conditions, the concentration of β-carotene was 19.13 mg/g DW and 6.60 mg/g DW by peel and paste, respectively. These conditions and the use of this methodology improved the extraction yield in mango BP's. UAE is an emerging technology that can be successfully used to obtain some bioactive compounds. The combination of a good experimental design with this technology can be useful in the industry to consider the use of BP's as potential food ingredients.

Nomenclature

βC	β-carotene
BHT	butylated hydroxytoluene
CSE	conventional solvent extraction
DW	dry weight

ET	extraction time
PC	pulse cycle
PDA	photodiode array detector
RSM	response surface methodology
SA	sonication amplitude
THF	tetrahydrofuran
UAE	ultrasound-assisted extraction

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