Vol. 16, No. 3 (2017) 845-859
Revista Mexicana de Ingeniería Química

# EFFECT OF ULTRASOUND ON THE CARBOHYDRATE EXTRACTION FROM SOTOL PLANTS (*Dasylirion wheeleri*) AT DIFFERENT POWERS AND TEMPERATURES

# EFECTO DEL ULTRASONIDO EN LA EXTRACCIÓN DE CARBOHIDRATOS A PARTIR DE PLANTAS DE SOTOL (*Dasylirion wheeleri*) A DIFERENTES POTENCIAS Y TEMPERATURAS

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Received April 28, 2017; Accepted June 9, 2017

## Abstract

Ultrasound-assisted extraction (UAE) of carbohydrates from powdered sotol plant (*Dasylirion wheeleri*) at different ultrasonic powers (UPs) (32.13-85.9 W) and temperatures (20.85-49.14 °C), in a 1:24 sotol:water ratio for 10 min was evaluated; a treatment control was done under thermal-traditional extraction. Total carbohydrates (TC), reducing sugars (RS), total fructan (FRU), glucose, fructose content, and average polymerization degree ($DP_n$) were evaluated. Fructan extraction kinetics and mass transfer coefficients ($K_L$) were calculated. RS and TC were significantly affected ($P < 0.05$) by temperature; FRU, by UP. Glucose, fructose, $DP_n$, and $K_L$ were affected ($P < 0.05$) by UP and temperature. TC and FRU reached maximum values at 54-59.0 W and 36.0 °C. $K_L$ reached a maximum at 55 W and 33.5 °C. The opposite trend was observed for glucose and fructose, presenting minimum values under these conditions, indicating minimal fructan damage. Under these conditions, $DP_n$ values ranged from 4.18 to 4.7 with maximum FRU of 7.97 g·100 $g^{-1}$ sotol d.m. Thermal treatment led to higher release of TC and RS and lower $DP_n$ compared with UAE, but showed similar FRU. UAE (54-59.05 W at 36.0 °C) can be used to obtain FRU from sotol heads.

*Keywords*: *Dasylirion*, carbohydrates, fructans, extraction, ultrasound.

## Resumen

Se evaluó el efecto del ultrasonido durante la extracción de carbohidratos a partir de sotol en polvo (*Dasylirion wheeleri*) a diferente potencia ultrasónica (PU) (32.13-85.9 W) y temperatura (20.85-49.14 ºC), en una relación de 1:24 sotol:agua durante 10 min; asimismo un tratamiento control fue realizado utilizando extracción térmica tradicional. En los extractos obtenidos se determinó el contenido de carbohidratos totales (CT), azúcares reductores (AR), fructanos totales (FRU), glucosa, fructosa y grados de polimerización promedio ($GP_n$). A partir de cinéticas de extracción de fructanos se calcularon los coeficientes de transferencia de masa ($K_L$). Los contenidos de AR y CT fueron afectados significativamente ($P < 0.05$) por la temperatura y los FRU por la PU. Los contenidos de glucosa y fructosa, así como $GP_n$ y $K_L$ fueron afectados ($P < 0.05$) tanto por la PU como por la temperatura. Los CT y FRU alcanzaron valores máximos en 54-59.05 W y 36.01 °C, $K_L$ generó un valor máximo en 55 W y 33.5 °C, sin embargo una tendencia opuesta fue observada para glucosa y fructosa, presentando un valor mínimo bajo las mismas condiciones, indicando un daño mínimo en los fructanos. Los valores de $GP_n$ oscilaron de 4.18 a 4.7 con un contenido máximo de fructanos de 7.97 g·100 $g^{-1}$ sotol b.s. El tratamiento térmico causó una mayor liberación de CT y AR con un menor $GP_n$ comparado con la extracción asistida con ultrasonido, pero con valores similares de fructanos. La extracción asistida con ultrasonido (54-59.05 W a 36.0 °C) puede ser usada para obtener fructanos a partir de piñas de sotol.

*Palabras clave*: *Dasylirion*, carbohidratos, fructanos, extracción, ultrasonido.

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# 1 Introduction

*Dasylirion* spp., commonly known as "sotol", is a perennial, succulent, polycarpic, and semi-cylindrical plant that produces a fibrous, shallow, and widely branching root (Bogler, 1998). The central part of the plant, known as the "head" or "pineapple", weighs 20-40 kg in mature plants (De la Garza-Toledo *et al*., 2008; Cruz-Requena *et al*., 2013). This desert plant grows in the wild, mainly in northern Mexico and the southern United States (Pardo-Rueda *et al*., 2015). *Dasylirion* spp. pineapples contain simple and complex carbohydrates such as glucose, fructose, and a series of fructooligosaccharides, which are fermented to produce a traditional distilled alcoholic beverage known as sotol (De la Garza-Toledo *et al*., 2008). Additionally, carbohydrates contained in sotol and other plants such as agave (Mancilla-Margalli and López, 2006) can be used to obtain fructan-type inulin or oligofructans, which can be used in the food industry as soluble fiber, fat, and sugar substitutes because of their physicochemical properties such as texture modification, gel formation, moisture retention, and food stabilization (Madrigal and Sangronis, 2007; Zhu *et al*., 2016). The chemical structure of fructans in this type of plant consists of a complex mixture of highly branched neo-fructans with both $\beta$-(2-1) and $\beta$-(2-6) linkages between fructose moieties (López *et al*., 2003; Mancilla-Margalli and López, 2006). This configuration gives fructans a prebiotic effect because they are resistant to enzymatic hydrolysis by human digestive enzymes, and therefore pass undigested into the colon where they are fermented by the colonic microflora (López and Urías-Silvas 2007; Leach and Sobolik, 2010). Fructans are found in the vacuoles of some plants along with glucose and fructose (Vijn and Smeekens, 1999). Thus, breakage of the cell wall may facilitate release of these components. However, breakage of some components of the cell wall, such as cellulose and hemicellulose, is complicated because these substances provide the cell wall with rigidity to prevent permeation. In addition, the pectic material that cements the cell wall prevents cell disruption (Raven, 1987; Sterling, 1963; Heredia-Léon *et al*., 2004). Traditionally, carbohydrates were extracted from plants by lixiviation with hot water (Bendjeddou *et al*., 2003; Dacome *et al*., 2005; Zha *et al*., 2009). This process is similar to those used for both beet sugar production (Franck, 2002) and inulin extraction from chicory roots (Franck, 2002; Roberfroid, 2004; Kelly, 2008). Extraction processes using hot water are employed because the elevated temperature increases the solubility of the components, particularly those of complex carbohydrates, increasing mass transfer during the extraction (Kim *et al*., 2001; Pardo-Rueda *et al*., 2015). However, these processes consume significant amounts of energy, and thus more sustainable options are required. In addition, thermal extraction under certain conditions may cause fructan hydrolysis, limiting its functionality for food applications (Madrigal and Sangronis, 2007; Zhu *et al*., 2016).

There is currently increasing interest in applying "green" or sustainable techniques that are more environmentally friendly (Chemat *et al*., 2017). Some physical methods (maceration, grinding, temperature, ultrasound, and pulsed electric field, among others) may promote cell disruption to release molecules such as fructans without affecting their physical and chemical properties. Ultrasound-assisted extraction (UAE) has been proposed for efficient extraction and can reduce the use of extraction solvents and processing time, therefore decreasing energy consumption (Li *et al*., 2013; Chemat *et al*., 2017). During UAE, high-intensity sound waves are transmitted through the liquid medium, resulting in alternating cycles of compression and rarefaction, leading to breakup of the liquid and generation of voids or cavities to form cavitation bubbles (Chemat *et al*., 2017). The bubbles grow to an equilibrium size and then collapse to generate energy for chemical and mechanical effects on the cell structure (Mason *et al*., 2003; O'Donnell *et al*., 2010). The mechanical effect in raw plant tissue disrupts the cell wall, facilitating solvent penetration into the cell and the release of biocomponents into the continuous phase (Toma *et al*., 2001; Lingyun *et al*., 2007; Ebringerová and Hromádková, 2010). Nevertheless, some previous studies (Lingyun *et al*., 2007; Apolinário *et al*., 2014) reported that application of ultrasound can break fructan molecules, forming low-molecular-weight fragments and diminishing the quality and functionality of these bioactive compounds. However, it is important to note that this operation depends on several factors, such as applied ultrasound power, sonication time, and temperature (Abou-Arab *et al*., 2011; Abozed *et al*., 2009; Paseephol *et al*., 2007; Pardo-Rueda *et al*., 2015). Recent studies have shown that during UAE for fructan from agave heads, it is possible to obtain higher fructan extraction rates at low temperatures (20-40 °C) and high

sonication powers, (Narváez-Flores *et al*., 2015). Similar results were reported by Pardo-Rueda *et al*. (2015) during carbohydrate extraction from sotol by thermo-sonication, showing the counteracting effect of the temperature over the sonication effect and minimizing the intensity of the sonication effect (Santos *et al*., 2009; Grönroos, 2010). Thus, it is important to evaluate the effect of this method in terms of better process conditions, mass transfer, and quality of the extracted compounds. The aim of this study was to evaluate the ultrasound-assisted extraction at different powers and temperatures on carbohydrates extraction from sotol plants.

## 2 Materials and methods

### 2.1 Material

The wild *Dasylirion wheeleri* head was collected in the winter season from Madera, Chihuahua area. The sotol heads used in this study were 8 years old with a weight of 8.5 kg and moisture content of 70%. The sotol head was stored at 1 °C at 90% relative humidity for 4 days before processing and physicochemical analysis in terms of diameter, weight, soluble solids content (°Brix), pH, and proximate analysis were performed according to AOAC methods (AOAC, 1998). Subsequently, the leafless head was peeled, cut into strips (0.05 m width × 0.01 m length), and dried using hot air at 70 ± 5 °C until a moisture level of approximately 5%. The dehydrated head was ground and passed through sieves to obtain uniform particles size of $5 \times 10^{-4}$ m (passed through 35-mesh and retained in 40-mesh). Next, the sotol powder was packed in hermetically plastic bags until analysis.

### 2.2 Carbohydrate extraction-assisted by ultrasound

Sotol powder (20 g) was subjected to UAE with a 1:24 (w/v) sotol:water ratio at different temperatures (20.85, 25, 35, 45, and 49.14 °C) and ultrasonic powers (32.13, 40, 59, 78, and 85.9 W) for 10 min, according to the experimental design shown in Tables 1 and 3. This extraction time was determined according to preliminary studies where the asymptotic behavior of extraction began. Ultrasound-assisted carbohydrate extraction experiments were carried out with a Branson Sonifier S-450 of 400 W with variable power (Branson Ultrasonics Sonifier, Danbury, CT) and 20 kHz of output frequency using a bath water for temperature control. The obtained extracts in each experimental combination were filtered under vacuum through Whatman No. 1 paper. Total carbohydrates (TC), reducing sugars (RS), total fructan (FRU), glucose, fructose content, and average degree of polymerization ($DP_n$) were determined. In addition, fructan extraction kinetics were performed for each experimental combination, where extract samples were periodically taken and FRU content was determined. These data were used for mass transfer coefficient ($K_L$) calculation. Thermal-traditional extraction conditions (90 °C at 40 min) were used as an experimental control.

### 2.3 Ultrasonic power calculation

The ultrasound power input into the fluid was calculated using the calorimetric method described by Narváez-Flores *et al*. (2015) and Moreno-Castro *et al*. (2015). First, the extraction medium temperature as a function of time was recorded under adiabatic conditions using a thermocouple. From the temperature versus time data collected, the initial temperature increase, dT/dt, was determined by linear fitting. The UP was determined according to Eq. (1)

$$UP = m \times C_p \left(\frac{dT}{dt}\right)_{t=0} \quad (1)$$

where $(dT/dt)$ is the change in temperature over time ($°C \cdot s^{-1}$), $C_p$ is the specific heat of extracted material at an average of 1.7 ± 0.01 °Brix (4.149 $kJ \cdot kg°C^{-1}$), calculated according to Asadi (2007), and m is the sample mass (kg). UP values were expressed in W.

### 2.4 Determination of total specific surface of sotol powder by Brunauer-Emmett-Teller method

Textural properties of sotol powder were characterized by nitrogen adsorption-desorption isotherms at 77 K using a commercial Autosorb-1C Quantacrome Instruments apparatus (Boynton Beach, FL, USA). Prior to each adsorption-desorption measurement, the samples, 1 g of sotol powder with a particle size of $5 \times 10^{-4}$ m, were introduced into the cell and degassed at $T$ = 473 K under $P = 10^{-2}$ mbar for 24 h. The obtained $N_2$ adsorption-desorption data were then analyzed according to the Brunauer-Emmett-Teller equation and Barrett-Joyner-Halenda equation for determination of the specific surface area and pore size distribution, respectively.

Table 1. Process variables and levels used in the experiment design.

| Process variables | Levels | | | | |
|---|---|---|---|---|---|
| | -1.41421 | -1 | 0 | +1 | +1.41421 |
| Ultrasonic power (W) | 32.13 | 40 | 59 | 78 | 85.9 |
| Temperature (°C) | 20.85 | 25 | 35 | 45 | 49.14 |

## 2.5 *Mass transfer coefficient ($K_L$)*

For each extraction kinetics, the mass transfer coefficients were obtained, considering that the change of the solute or sugars ($N$) with respect to time ($t$) is described by Eq. (2):

$$\frac{dN}{dt} = K_L a[C_S - C] \tag{2}$$

where $K_L a$ represents the overall mass-transfer coefficient across an effective external surface area ($m^3 \cdot s^{-1}$), and $a$ ($m^2$), C ($g \cdot 100\ g^{-1}$), and $C_s$ ($g \cdot 100\ g^{-1}$) represent the concentrations of carbohydrate in the solvent at any time, t (s), and at saturation condition, respectively (Heldman and Singh, 1981; Geankoplis, 1998). If the solid-liquid extraction is carried out in a batch process where the total volume, $V$, of solution ($m^3$) is kept constant, the following expression is obtained:

$$dN = Vd \tag{3}$$

This leads to Eq. (4):

$$C = C_s\left[1 - \exp\left(-\frac{K_L a}{V}t\right)\right] \tag{4}$$

Equation 4 implies that the volumetric mass-transfer coefficient ($s^{-1}$) influences the rate at which the solvent approaches the saturation concentration. From the extraction kinetics of fructans for each treatment, the slopes $(K_L a)/V$ were obtained. The process was batch to volume ($V$) of 0.0480 $m^3$ and with the determined surface area ($a$), the mass transfer coefficient ($K_L$) was obtained for each treatment.

## 2.6 *Analytical methods*

The moisture, protein, fat, fiber, and ash contents of the sotol heads were measured according to AOAC (1998) methods 950.02, 960.52, 920.39, 962.09, and 923.03, respectively; and the carbohydrate contents were then obtained by calculating the difference in mass between the total mass and sum of the other components. Total soluble solids (°Brix) were measured using an Abbe hand refractometer (Atago Co. Ltd., Tokyo, Japan) according to AOAC method 932.12 and the pH was determined using a digital pH meter (pH 211, Hanna Instruments, Ann Arbor, MI, USA) according to AOAC method 981.12 (AOAC, 1996). Total sugar content of the sotol extracts was measured using the phenol-sulfuric acid method (Dubois *et al*., 1956) and reducing sugar content was measured using the dinitrosalicylic method (Miller, 1959). D-Fructose was used as a standard in both cases. The FRU content was determined using a fructan assay kit (Megazyme, Wicklow, Ireland) as described by McCleary *et al*. (2000) and Narváez-Flores *et al*. (2015) and was expressed as $g \cdot 100\ g^{-1}$ dry matter (d.m.) of sotol.

## 2.7 *High-performance anion exchange chromatography (HPAEC-PAD) profile of sotol extracts*

The separation of fructans from sotol powder extracts was performed by high-performance anion exchange coupled with pulsed amperometric detection (HPAEC-PAD) on a Thermo Scientific Dionex ICS-5000$^+$ system (Waltham, MA, USA) equipped with an electrochemical detector with a gold working electrode and AgCl reference electrode. Before characterization, the extracts were passed through strong anionic and cationic resin exchangers, after which they were diluted to the appropriate concentration with deionized water. After filtration through a 0.45-$\mu$m membrane, 25 $\mu$L of sample was injected into the system using an autosampler. The sample was injected through an analytical CarboPac PA-100 column (4 × 250 mm) with a guard column (Thermo Scientific). The column temperature was 30 °C and a flow rate of 1 mL/min was applied. The eluents used for fructan separation were (A) 150 mM NaOH and (B) 1 M sodium acetate diluted in 150 mM NaOH. The elution gradient was 0-10 min with 100% eluent A, 10-85 min with a linear gradient from 0 to 45% eluent B, and 85-90 min with 45% to 0% eluent B. Glucose, fructose, sucrose, 1-kestose (DP 3), 1,1-kestotetraose (DP 4), 1,1,1-kestopentaose (DP 5), and inulin from chicory were used as standards.

Table 2. Physicochemical properties of wild *Dasylirion wheeleri* plant.

| Properties | Fresh sotol | Dehydrated sotol |
|---|---|---|
| Diameter (m) | 0.827 ± 0.011 | —- |
| Weight (kg) | 8.50 ± 0.228 | —- |
| ºBrix | 12.33 ± 0.329 | —- |
| pH | 5.39 ± 0.508 | —- |
| Specific surface area ($m^2\ g^{-1}$) | —- | 0.4019 ± 0.008 |
| Proximate composition (%) | | |
| Moisture | 76.64 ± 0.03 | 6.34 ± 0.04 |
| Crude fiber | 6.99 ± 0.22 | 32.25 ± 1.05 |
| Ash | 2.19 ± 0.55 | 4.05 ± 0.04 |
| Protein | 0.86 ± 0.04 | 3.15 ± 0.06 |
| Fat | 0.64 ± 0.10 | 2.12 ± 0.02 |
| Carbohydrates | 12.68 ± 0.06 | 52.09 ± 0.08 |

*Mean ± standard error.

## 2.8 *Hydrolysis of sotol extracts*

Extract hydrolysis was performed according to the method described by Ronkart *et al*. (2007) with some modifications. The sotol extract (1 mL) was adjusted to pH 4.5 by the addition of sodium acetate buffer (9 mL) to create optimal conditions for the fructanase enzyme. 50 $\mu$L of fructanase mixture (Megazyme Wicklow, Ireland) comprising exo-inulinase (2000 $U{\cdot}mL^{-1}$) and endo-inulinase (65 $U{\cdot}mL^{-1}$) was added to each extract, after which the samples were hydrolyzed at 50 °C with agitation for 24 h and then boiled for 10 min to inactivate the enzyme. The final product of the enzymatic reaction was diluted to the appropriate concentration and filtered through a 0.45-$\mu$m membrane before being injected into the HPAEC-PAD system for glucose and fructose quantification.

## 2.9 *Determination of the average degree of polymerization ($DP_n$)*

The $DP_n$ of the sotol extracts were determined as described by López-Molina *et al*. (2005), where $DP_n$ corresponds to the total number of glucose and fructose units in an inulin sample divided by the total number of inulin molecules, excluding the glucose, fructose, and sucrose units present in the initial sample. Glucose and fructose levels were quantified using HPAEC-PAD analysis before and after total hydrolysis of the extracts. By calculating the fructose:glucose ratio, the $DP_n$ was determined as the number of fructose units per number of glucose units plus one according to Eq. (5):

$$DP_n = \frac{F_{final} - F_{start}}{G_{final} - G_{start}} + 1 \tag{5}$$

where $F_{start}$ and $F_{end}$ refer to the fructose concentrations before and after total hydrolysis, respectively, and $G_{start}$ and $G_{end}$ refer to the glucose concentrations before and after total hydrolysis, respectively.

## 2.10 *Experimental design and statistical analysis*

A second-order rotatable central composite design was used and the results were analyzed using response surface methodology (Myers, 1971). The UP and extraction temperature were independent variables. The fitted second-order polynomial is given by Eq. (6):

$$Y_i = b_o + b_1X_1 + b_2X_2 + b_{11}X_1^2 + b_{22}X_2^2 + b_{12}X_1X_2 + \varepsilon \tag{6}$$

where $Y_i$ is the predicted response for the experiment, $X_1$ is the ultrasonic power value for the experiment i; $X_2$ is the extraction temperature for the experiment i; and $b_0$, $b_1$, $b_2$, $b_{12}$, $b_{11}$, and $b_{22}$ are regression coefficients of the intercept, linear effects, squared effects, and interactions, respectively, and $\varepsilon$ is the error. The levels and combinations of the variables are shown in Tables 1 and 3, respectively. Design Expert software v. 6.01 (Stat-Ease, Inc. 2005, Minneapolis, MN, USA) was used to fit the response surface models to the experimental data and Minitab v. 16 software (Minitab, Inc., State College, PA, USA) was used to

Table 3. Total carbohydrates, reducing sugars, total fructan content, mass transfer coefficient, glucose, fructose, and average degree of polymerization of extracts from sotol (*Dasylirion wheeleri*) obtained at different ultrasonic powers and temperatures.

| Treatment | UP (W) | T (ºC) | TC (g·100 g$^{-1}$ d.m.) | RS (g·100 g$^{-1}$ d.m.) | FRU ( g·100 g$^{-1}$ d.m.) | $K_L$ (m·s$^{-1}$) | Glucose (g·100 g$^{-1}$ d.m.) | Fructose (g·100 g$^{-1}$ d.m.) | DP$_n$ |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 40 | 25 | 24.92 ± 0.81$^{bc}$ | 12.95 ± 0.61$^{e}$ | 7.31 ± 0.05$^{c}$ | 4.08×10$^{-7}$ ± 1.2×10$^{-8}$ | 3.53 ± 0.06$^{bcd}$ | 4.68 ± 0.05$^{a}$ | 7.52 ± 0.74$^{c}$ |
| 2 | 78 | 25 | 22.51 ± 1.29$^{d}$ | 12.70 ± 0.09$^{e}$ | 7.54 ± 0.08$^{abc}$ | 3.41×10$^{-7}$ ± 2.4×10$^{-8}$ | 3.84 ± 0.02$^{a}$ | 4.80 ± 0.02$^{a}$ | 5.87 ± 0.15$^{d}$ |
| 3 | 40 | 45 | 23.69 ± 0.39$^{cd}$ | 17.06 ± 0.22$^{b}$ | 7.45 ± 0.52$^{bc}$ | 3.10×10$^{-7}$ ± 1.9×10$^{-8}$ | 3.44 ± 0.03$^{cde}$ | 3.75 ± 0.22$^{cd}$ | 9.17 ± 0.59$^{ab}$ |
| 4 | 78 | 45 | 22.38 ± 0.75$^{d}$ | 17.16 ± 0.36$^{b}$ | 7.34 ± 0.31$^{bc}$ | 3.12×10$^{-7}$ ± 2.3×10$^{-8}$ | 3.21 ± 0.02$^{fg}$ | 4.60 ± 0.05$^{ab}$ | 5.03 ± 0.42$^{de}$ |
| 5 | 32.13 | 35 | 24.44 ± 1.10$^{bc}$ | 14.96 ± 0.17$^{d}$ | 7.45 ± 0.37$^{bc}$ | 4.39×10$^{-7}$ ± 2.9×10$^{-8}$ | 3.81 ± 0.05$^{a}$ | 4.53 ± 0.16$^{a}$ | 10.56 ± 0.67$^{a}$ |
| 6 | 85.9 | 35 | 25.68 ± 0.78$^{b}$ | 15.90 ± 0.34$^{c}$ | 7.42 ± 0.31$^{bc}$ | 2.97×10$^{-7}$ ± 1.6×10$^{-8}$ | 3.67 ± 0.04$^{ab}$ | 4.76 ± 0.59$^{a}$ | 5.61 ± 0.59$^{de}$ |
| 7 | 59 | 20.85 | 20.74 ± 0.20$^{e}$ | 10.47 ± 0.25$^{f}$ | 7.77 ± 0.60$^{abc}$ | 3.77×10$^{-7}$ ± 3.9×10$^{-8}$ | 3.35 ± 0.02$^{def}$ | 4.09 ± 0.04abc | 7.79 ± 0.72$^{bc}$ |
| 8 | 59 | 49.14 | 24.86 ± 0.63$^{bc}$ | 16.77 ± 0.62$^{b}$ | 7.94 ± 0.10$^{abc}$ | 3.34×10$^{-7}$ ± 2.2×10$^{-8}$ | 3.26 ± 0.02$^{efg}$ | 4.59 ± 0.09$^{ab}$ | 5.37 ± 0.49$^{de}$ |
| 9 | 59 | 35 | 25.71 ± 0.60$^{b}$ | 16.06 ± 0.60$^{bc}$ | 7.93 ± 0.18$^{ab}$ | 5.32×10$^{-7}$ ± 6.0×10$^{-8}$ | 3.16 ± 0.07$^{g}$ | 3.40 ± 0.07$^{d}$ | 4.73 ± 0.43$^{e}$ |
| 10 | 59 | 35 | 25.94 ± 0.42$^{b}$ | 17.06 ± 0.39$^{bc}$ | 8.12 ± 0.07$^{ab}$ | 5.18×10$^{-7}$ ± 2.5×10$^{-8}$ | 3.11 ± 0.01$^{g}$ | 3.33 ± 0.03$^{d}$ | 4.44 ± 0.15$^{e}$ |
| 11 | 59 | 35 | 26.27 ± 0.43$^{b}$ | 15.84 ± 0.11$^{bc}$ | 8.02 ± 0.15$^{ab}$ | 5.56×10$^{-7}$ ± 4.1×10$^{-8}$ | 3.26 ± 0.02$^{g}$ | 3.51 ± 0.01$^{d}$ | 4.62 ± 0.09$^{e}$ |
| 12 | 59 | 35 | 25.72 ± 0.64$^{b}$ | 16.92 ± 0.16$^{bc}$ | 7.96 ± 0.24$^{ab}$ | 5.27×10$^{-7}$ ± 6.3×10$^{-8}$ | 3.15 ± 0.01$^{g}$ | 3.38 ± 0.02$^{d}$ | 4.73 ± 0.04$^{e}$ |
| 13 | 59 | 35 | 24.98 ± 0.64$^{b}$ | 17.02 ± 0.61$^{bc}$ | 7.97 ± 0.16$^{ab}$ | 5.05×10$^{-7}$ ± 1.5×10$^{-8}$ | 3.08 ± 0.01$^{g}$ | 3.34 ± 0.02$^{d}$ | 4.37 ± 0.06$^{e}$ |
| SE | – | – | 0.3578 | 0.1442 | 0.0471 | 1.298 ×10$^{-8}$ | 0.0277 | 0.0748 | 0.1997 |
| Control | 0 | 90 | 30.95 ± 0.71$^{a}$ | 20.27 ± 0.35$^{a}$ | 8.26 ± 0.29$^{a}$ | — | 3.61 ± 0.05$^{bc}$ | 3.94 ± 0.07$^{bc}$ | 3.65 ± 0.33$^{f}$ |

*Values presented are the average of triplicate measurements ± standard deviation. UP, ultrasonic power; T, temperature; TC, total carbohydrates; RS, reducing sugars; FRU, total fructans; $K_L$, mass transfer coefficient; DP$_n$, average degree of polymerization, SE, standard error.

compare the means between treatments by Tukey´s test. Significant differences were defined as $P < 0.05$.

## 3 Results and discussion

### 3.1 Physicochemical characterization of sotol plants

Size dimensions (diameter and weight) as well as pH, °Brix, and proximate composition of wild sotol head fresh and specific surface area of dehydrated powder are shown in Table 2. This composition of the sotol head is similar to that reported by Cruz-Requena *et al*. (2013) in sotol (*Dasylirion cedrosanum*) and for some Agavaceas (Narváez-Flores *et al*., 2015; Flores-Girón *et al*., 2016). Regarding the specific surface area, it was observed that the sotol powder showed a very small hysteresis (Fig. 1). Hysteresis results from interaticular spaces of sotol powder and not from the presence of other type of pores in the material. For this reason, sotol powder has a low surface area (Table 2), indicating that it is a non-porous material (Hu and Srinivasan, 1999). This low value of surface area could not influence mass transfer during the extraction process. A typical value for the vegetal material was reported by Guevara-Amatón *et al*. (2010) for nutshell (0.708 m$^2$·g$^{-1}$) and high values in activated carbon from almond and coconut shell were reported by Marcilla *et al*. (2000) and Hu and Srinivasan (1999), respectively.

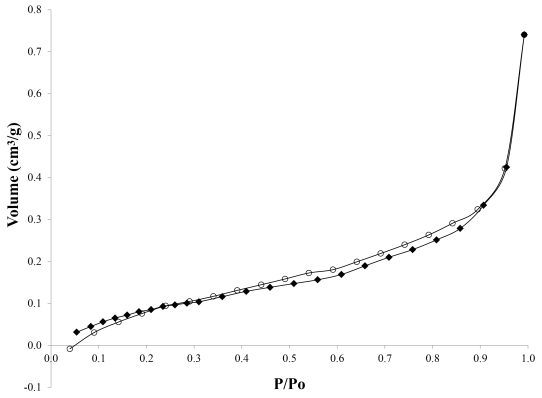


Fig. 1. Adsorption and desorption isotherms of the sotol powder. ♦ Desorption. o Adsorption.

Some studies showed that many biopolymers result in large sorption surface due to the existence of an intrinsic microporous structure in the materials (Aguerre *et al*., 1989). Additionally, these differences result from the number and size of pores in the carbohydrate matrix, which determine the total sorption area. Thus, the rate and extent of hydration of the materials are influenced by the surface properties of the pores (Sing, 2001; Rosa *et al*., 2010).

### 3.2 Model fitting

The influence of UP and extraction temperature on carbohydrate extraction is shown in Table 4. Analysis of variance for all responses indicated adequate adjustment without a significant lack of fit, except for

Table 4. Analysis of variance of total carbohydrates, reducing sugars, total fructan content, mass transfer coefficient, glucose, fructose, and average degree of polymerization of extracts from sotol (*Dasylirion wheeleri*).

| | | Mean squares | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Source | DF | TC | RS | FRU | $K_L$ | Glucose | Fructose | $DP_n$ |
| Model | 5 | 4.29 | 10.46* | 0.16* | $2.178\times10^{-14}$* | 0.15* | 0.80* | 8.51* |
| A-Ultrasonic power | 1 | 0.48 | 0.17 | $8.858\times10^{-4}$ | $8.895\times10^{-15}$* | $1.876\times10^{-3}$ | 0.21 | 20.46* |
| B-Temperature | 1 | 2.5 | 38.24* | $4.357\times10^{-3}$ | $4.419\times10^{-15}$* | 0.087* | 0.021 | 0.86 |
| AB | 1 | 0.3 | 0.031 | 0.031 | $1.193\times10^{-15}$ | 0.073* | 0.14 | 1.55 |
| $A_2$ | 1 | 1.5 | 1.57a | 0.73* | $4.948\times10^{-14}$* | 0.57* | 2.63* | 17.06* |
| $B_2$ | 1 | 17.69* | 13.25* | 0.089 | $5.718\times10^{-14}$* | 0.032 | 1.47* | 4.63* |
| Residual | 7 | 1.67 | 0.27 | 0.028 | $7.205\times10^{-16}$ | 0.01 | 0.071 | 0.52 |
| Lack of fit | 3 | 3.60* | 0.23 | 0.042 | $1.207\times10^{-15}$ | 0.018 | 0.16* | 0.93 |
| Pure error | 4 | 0.23 | 0.31 | 0.017 | $3.554\times10^{-16}$ | $5.145\times10^{-3}$ | $5.714\times10^{-3}$ | 0.21 |
| $R^2$ | | 0.6468 | 0.9646 | 0.805 | 0.956 | 0.9093 | 0.8893 | 0.9213 |

*Significance at $P < 0.05$. TC, total carbohydrates; RS, reducing sugars; FRU, total fructans; $K_L$, mass transfer coefficient; $DP_n$, average degree of polymerization.

Table 5. Regression coefficients of second-order model of relationships between variables for extracts from sotol (*Dasylirion wheeleri*).

| Coefficients | TC | RS | FRU | $K_L$ | Glucose | Fructose | $DP_n$ |
|---|---|---|---|---|---|---|---|
| $b_0$ | 25.71 | 16.61 | 7.97 | $5.278\times10^{-7}$ | 3.17 | 3.4 | 4.74 |
| $b_1$ | -0.24 | 0.15 | 0.011 | $-3.335\times10^{-8}$* | -0.015 | 0.16 | -1.60* |
| $b_2$ | 0.56 | 2.19* | 0.023 | $-2.350\times10^{-8}$* | -0.10* | -0.051 | -0.33 |
| $b_{12}$ | 0.28 | 0.088 | -0.088 | $1.727\times10^{-8}$ | -0.13* | 0.18 | -0.62 |
| $b_{11}$ | -0.46 | -0.47 | -0.032* | $-8.433\times10^{-8}$* | 0.29* | 0.61* | 1.57* |
| $b_{22}$ | -1.59* | -1.38* | -0.11 | $-9.066\times10^{-8}$* | 0.068 | 0.46* | 0.82* |

[a] Significance at $P < 0.05$. TC, total carbohydrates; RS, reducing sugars; FRU, total fructans; $K_L$, mass transfer coefficient; $DP_n$, average degree of polymerization.

TC content. Regression coefficients were obtained by fitting the experimental data to the second-order model (Table 5). Models showed satisfactory $R^2$ values, except for the TC content fitted model.

### *3.3 Total carbohydrate (TC) extraction*

TCs include soluble sugars extracted during the UAE process. Extraction temperature had a significant effect ($P < 0.05$) on TC extraction (Table 4). However, the second-order model did not adequately predict the data behavior ($R^2 = 0.6468$), showing a lack of fit ($P < 0.05$). The effects of temperature and UP conditions on TC extraction are shown in Figure 2. Increases in temperature increase the TC content, reaching a maximum value of 25.78 g·100 $g^{-1}$ sotol d.m. at a sonication power of 54.87 W and extraction temperature of 36.5 °C. Despite these results, the TC contents obtained by UAE (Table 3) were lower ($P < 0.05$) than those obtained by thermal-traditional treatment (30.95 g·100 $g^{-1}$ sotol d.m.). The trend presented (Fig. 2a) during UAE showed increases in TC contents as temperatures increased, reaching the maximum value described above. This may be because sugar solubility is favored by temperature increases (Pardo-Rueda *et al*., 2015; Narváez-Flores *et al*., 2015). However, additional temperature increases caused a decrease in TC content. Some reports showed that temperature increases minimize the cavitation effect (Grönroos, 2010; Pardo-Rueda *et al*., 2015), diminishing the extraction of components, as observed in this study. The TC observed in this study was higher than that reported by Pardo-Rueda *et al*. (2015) who conducted sonothermal extraction at 40 °C from sotol (*Dasylirion leiophylum*) plants.

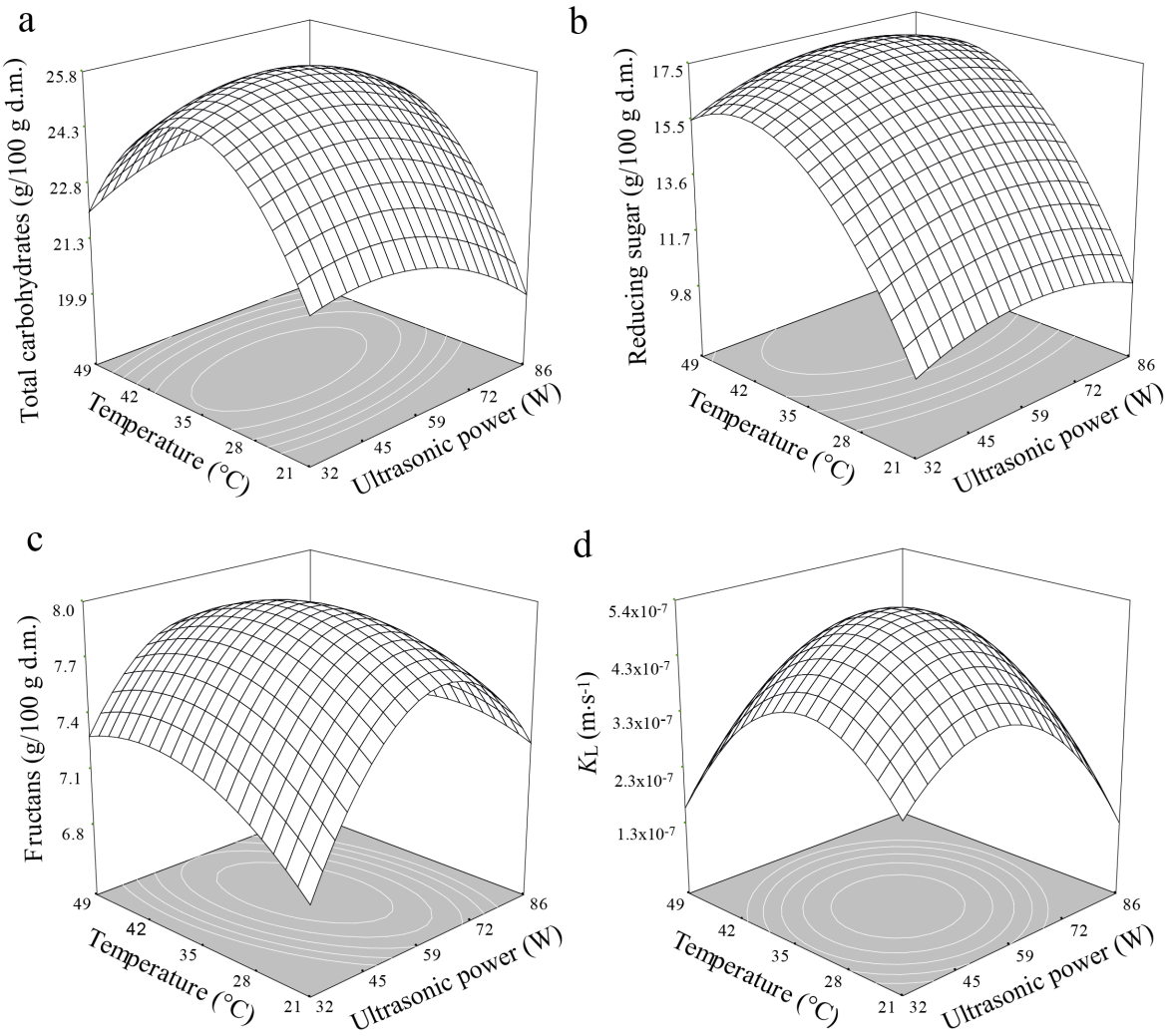


Fig. 2. Response surface for the effect of ultrasonic power and extraction temperature on total carbohydrates (a), reducing sugars (b), total fructan content (c), and mass transfer coefficient (d) of *Dasylirion wheeleri* extracts.

### *3.4 Reducing sugar (RS) extraction*

Reducing sugar extraction was significantly affected ($P < 0.05$) by the UP and temperature. The second-order model was adequately adjusted to obtain an $R^2$ of 0.9646 (Table 4) and topography of maximum value of 17.49 g·100 g$^{-1}$ sotol d.m. for UP and temperature conditions of 63.36 W and 43 °C, respectively. Fig. 2b represents the RS behavior at different levels of temperature and UP, showing that as these variables were increased, RS values also increased, reaching a maximum value under the conditions described above. RS contents obtained in all UAE treatments resulted in lower values ($P < 0.05$) than thermal-traditional treatment (20.27 g·100 g$^{-1}$ sotol d.m.). This indicates greater hydrolysis of complex polysaccharides by high temperatures during thermal extraction. Similar results were reported during the sonothermal and thermal extraction of carbohydrates from sotol at 40 °C, with higher RS release by thermal treatments compared with sonothermal treatment at 40 °C (Pardo-Rueda *et al.*, 2015).

### *3.5 Total fructan (FRU) extraction*

Fructans are polymers composed of fructose units. Fructan extraction was significantly affected by UP in the quadratic term ($P < 0.05$). The model was adequately adjusted to the experimental data, giving an $R^2 = 0.8050$ (Table 4) and the model-adjusted coefficients shown in Table 5.

The effects of both independent variables on FRU extraction are shown in Fig. 2c, with values ranging from 7.31 to 8.12 g·100 $g^{-1}$ sotol d.m. Increases in UP resulted in increased fructan content, reaching a maximum value of 7.97 g·100 $g^{-1}$ sotol d.m. at 36 °C and 59.05 W. The maximum FRU content obtained in sonication treatment was similar ($P > 0.05$) to that for thermal-traditional treatment, without showing an improvement in FRU extraction. However, UAE treatment required lower temperatures and shorter processing times (10 min). Increases or decreases in UP decreased fructan extraction (Fig. 2c). This minimal contribution of the extraction temperature to fructan content was reported by Narváez-Flores *et al*. (2015) during extraction of fructans from *Agave tequilana* Weber var. azul; despite obtaining a significant effect for extraction temperature, this was not reflected in the extraction of these bioactive compounds. For the temperature range evaluated (20-40 °C), limited fructan solubility was observed in water except at 40 °C, which is close to the solubility limit of these components. A similar trend was observed in this study; temperature did not have a large effect on fructan extraction, but was close to the solubility limit of these components, and thus extraction yielded maximum values at temperatures close at 40 °C. Extraction conditions at higher temperatures lead to a decrease in fructan extraction. This can be attributed to a counter-effect of the cavitation mechanism, which minimizes the extraction of components (Santos *et al*., 2009; Grönroos, 2010; Pardo Rueda *et al*., 2015; Chemat *et al*., 2017).

### *3.6 Mass transfer coefficient ($K_L$) during fructan extraction*

The mass transfer coefficient ($K_L$) for fructan extraction indicated that the extraction rate of these components was dependent on the experimental conditions (Table 3). Table 4 shows that temperature and UP has significant linear and quadratic effects ($P < 0.05$). The model showed a satisfactory adjustment ($R^2 = 0.9560$) in the description of the fructan transfer coefficient ($K_L$) during UAE. Increases in temperature and UP caused an increase in $K_L$ values, reaching a maximum value of $9.33 \times 10^{-7}$ m·$s^{-1}$ at 33.5 °C and 54.96 W (Fig. 2d). Experimental conditions outside this range led to a decrease in $K_L$ values. The same conditions for UP and temperature at which $K_L$ reached a maximum were similar to the optimal conditions for maximum fructan extraction. These results agree with recent studies showing that UP significantly affects the volumetric mass transfer coefficient during fructan extraction from agave, with higher extraction rates obtained at low temperatures and high powers (Narváez-Flores *et al*., 2015). These results indicate that ultrasound causes changes and ruptures in the cell wall, favoring the transfer of carbohydrates into the extraction medium (Ebringerová and Hromádková, 2010). However, the use of higher temperatures does not result in significant improvements in fructan extraction. As described above, this is because high temperatures counteract the effect of cavitation (Grönroos, 2010; Pardo Rueda *et al*., 2015).

### *3.7 Glucose content*

Glucose is a reducing sugar that is released during the UAE of carbohydrates. Glucose values in the experimental range oscillated between 3.08 and 3.84 g·100 $g^{-1}$ sotol d.m. (Table 3). Higher values of temperature and UP result in a decrease in glucose content (Fig. 3a), showing a minimum at 64.19 W and 45.5 °C with a value of 3.10 g·100 $g^{-1}$ sotol d.m, while combinations of both variables (high temperature-low UP and high UP-low temperature) result in high glucose contents. These changes are caused by the combined effect of sonication and temperature. Sonication may rupture cell wall components and release fructan simple sugar as glucose under certain conditions, as reported by Toma *et al*. (2001) and Pardo-Rueda *et al*. (2015). While temperature increases cause an increase of glucose solubility. Similar results were reported by Pardo-Rueda *et al*. (2015) during thermal extraction of carbohydrates from sotol; glucose content increased in extractions performed from 40 °C to 70 °C.

### *3.8 Fructose content*

Fructose is the main structural unit of fructans, which was significantly affected ($P < 0.05$) by the quadratic terms of temperature and UP (Table 4). The model estimated the variability of the data by 88.9% and showed a minimum value of fructose (3.38 g·100 $g^{-1}$ sotol d.m.) at 56.3 W and 36 °C (Fig. 3b). Most fructose values obtained by UAE treatment were similar ($P > 0.05$) to those obtained by thermal treatment (3.94 g·100 $g^{-1}$ sotol d.m.), except for the highest UP treatments (Table 3) which showed the highest fructose contents ($P < 0.05$). This indicates that in both treatments, some hydrolysis of complex polysaccharides occurs.

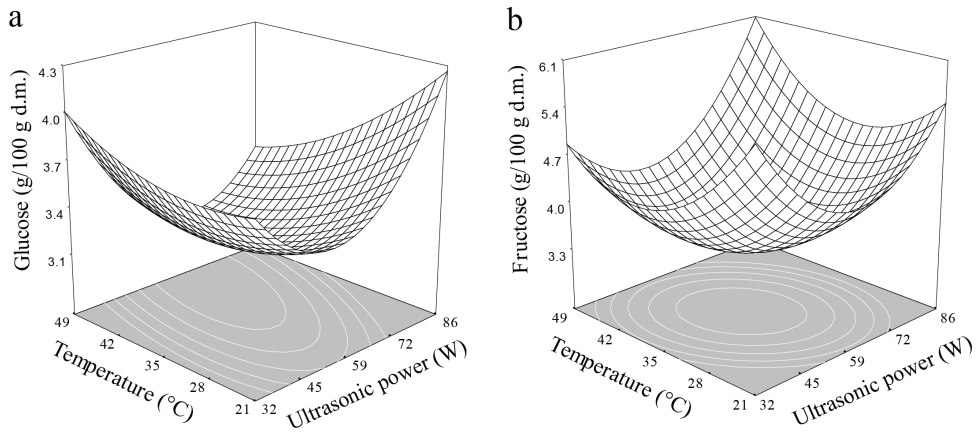


Fig. 3. Response surface for the effect of ultrasonic power and extraction temperature on glucose (a), and fructose (b) contents of *Dasylirion wheeleri* extracts.

Similarly, the presence of fructose in the extracts by sonication treatments not only ruptured the cell wall, as described Narváez-Flores *et al*. (2015), but also caused breakage of fructan molecules, releasing simple sugars such as fructose and glucose. Additionally, this result is consistent with those of fructan extraction (Fig. 2c), in which under similar UP and temperature conditions, minimum fructose release and maximum fructan content were obtained.

### *3.9 HPAEC-PAD profiles and average degree of polymerization ($DP_n$) of fructans*

The sotol extracts analyzed by HPAEC-PAD, revealed according to the stardards of individual sugars and inulin from chicory (Fig. 4c), that in treatments of Fig. 4a-d, and the treatment of thermal-traditional extraction (Fig. 4e-f) the fructooligosaccharides 1-kestose (DP3), 1,1-kestotetraose (DP4), and DP5 (1,1,1-kestopentaose) (DP5) (standard with higher DP used in this study) were identified. Likewise, subsequent peaks could be observed, indicating the presence of DPs greater than 5 units of fructose. Identification of these fructooligosaccharides had been reported by Alvarado *et al*. (2014) and Chávez-Rodríguez *et al*. (2016) in spray dried fructans of *A. tequilana*. Despite the identification of some peaks using the standards for the separation and identification of fructans from sotol extracts, as can be seen in Fig. 4 a clear difference between sonicated treatments (Fig. 4a-d) and thermal-traditional treatment was not observed. The sotol profiles found in all treatments were similar to those reported by López *et al*. (2003), Mancilla-Margalli and López (2006), Waleckx *et al*. (2008), and Chávez-Rodríguez *et al*. (2016) for *A. tequilana* Weber var azul. As can be observed these chromatograms were not as clear as those obtained for chicory inulin (Figure 4g-h). This is because, the identification of fructans with long DPs for *D. wheeleri* in this study is difficult; as reported by Arrizon *et al*. (2010) and Waleckx *et al*. (2008) in fructans of *A. tequilana*. This is attributed to fructans of this type of plants exist as a complex mixture of highly branched neo-fructans with $\beta$-(2-1) and $\beta$-(2-6) linkages between fructose moieties (López *et al*., 2003; Mancilla-Margalli and López, 2006), which is more complex than the simple inulin series (linear $\beta$-(2-1)-linked series) from chicory. For this reason, DPs which, reflect the number of structural units (fructoses) forming a fructan molecule were quantified according to López-Molina *et al*. (2005) as $DP_n$, and these results are shown in Table 3.

Although with HPAEC-PAD profiles no clear differences were observed between treatments and with thermal-traditional treatment, in Table 3 is observed the effect of UP in $DP_n$. This response was dependent on the linear and quadratic effect of UP and temperature in the quadratic effect ($P < 0.05$) (Table 4). The second-order model was adjusted to explain the data variability by 92.13% (Table 4). $DP_n$ of different extracts showed values ranging from 4.37 to 10.56, founding that at high UP the $DP_n$s found were lower and singnificantly different ($P < 0.05$) to treatments extracted at lower UP (32.13 and 40 W). The surface topography of this response yielded a minimum value of 4.18 at 39.3 °C and 70.32 W (Fig. 5).

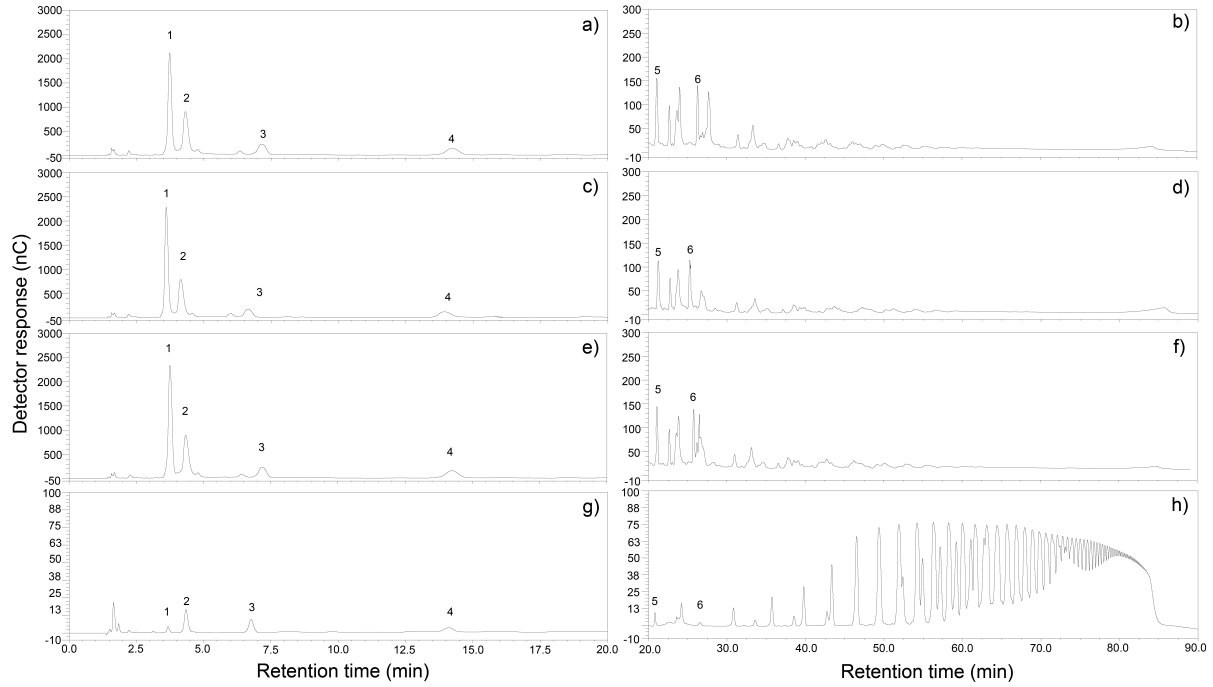


Fig. 4. HPAEC-PAD profile of fructans obtained from *Dasylirion wheeleri* plants extracted at different ultrasonic powers and extraction temperatures. (a-b) 32.13 W and 35 °C, (c-d) 85.9 W and 35 °C, (e-f) control without ultrasound at 90 °C, and (g-h) inulin from chicory. Peaks 1, 2, 3, 4, 5, and 6 were identified by comparison with standards of known retention time and correspond to glucose, fructose, sucrose, 1-kestose, 1,1-kestotetraose, and 1,1,1-kestopentaose, respectively.

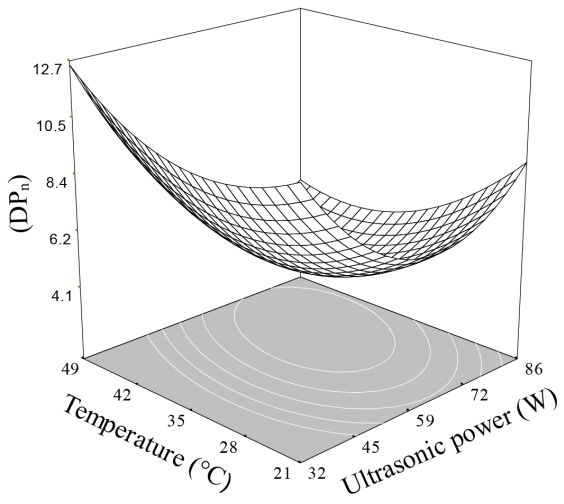


Fig. 5. Response surface for the effect of ultrasonic power and extraction temperature on the average degree of polymerization ($DP_n$).

In general, $DP_n$ values found in extracts obtained by sonication were higher than those obtained by thermal-traditional extraction (3.65), with high $DP_n$ values ($P < 0.05$) in UAE treatments with lower UP (Table 3). These results indicate that high UP causes breakage of fructan molecules, resulting in extracts with low DP. High UP can degrade fructans via glycosidic bond cleavage in both structural and branched polysaccharides (Ebringerová and Hromádková, 2010; Pardo-Rueda *et al*., 2015). Similar findings have been reported by Lingyun *et al*. (2007). The lowest $DP_n$ value (3.65) of the thermal-traditional treatment, indicates more fructan degradation. Therefore, in terms of fructan degradation, the UAE process represents an improved method for the fructans extraction from sotol plants compared with the traditional method.

## Conclusions

The proposed second-order model, involving the response surface methodology, adequately predicted fructan extraction assisted by ultrasound from wild sotol (*D. wheeleri*), at different UPs and temperatures. An increase in UP up to 59.05 W and 36.01 °C leads to an increase in fructan content, with minimal simple sugar release (glucose and fructose), indicating minimum fructan hydrolysis. This resulted in extracts with $DP_n$ values of 4.7. Although fructan content did not differ from those obtained by thermal-traditional extraction, UAE minimized damage to the sugar molecule, resulting in fructans with higher $DP_n$ at short extraction times. These results suggest that UAE is a friendly alternative with lower energy consumption for fructan extraction from plant tissues such as sotol (*D. wheeleri*).

### *Acknowledgements*

The authors acknowledge the National Council of Science and Technology (CONACYT-Mexico) for financial support via a Basic Research Project Grant (no: 182169) and Convocatoria de Ciencia Básica SEP-CONACYT 2012.

### *Nomenclature*

| | |
|---|---|
| UAE | ultrasound assisted extraction |
| TC | total carbohydrates, g·100 $g^{-1}$ |
| RS | reducing sugars, g·100 $g^{-1}$ |
| FRU | total fructan content, g·100 $g^{-1}$ |
| $DP_n$ | average degree of polymerization |
| $K_L$ | mass transfer coefficient, m·$s^{-1}$ |
| UP | ultrasonic power, W |
| T | temperature, ºC |
| $dT/dt$ | change in temperature over time, °C·$s^{-1}$ |
| $C_p$ | specific heat, kJ·$kg^{-1}$·°$C^{-1}$ |
| $m$ | mass, kg |
| $a$ | area, $m^2$ |
| $V$ | volume, $m^3$ |
| d.m. | dry matter |

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