Vol. 23, No. 2(2024) Alim24237
Revista Mexicana de Ingeniería Química

Assisted (ultrasound or high shear impeller) soybean oil/lecithin extraction of polyphenolic compounds from red cactus pear peel: Extracts effects on oleogels properties

Extracción con aceite de soya/lecitina asistida (ultrasonido o un impulsor de alto corte) de compuestos polifenólicos de cáscara de tuna roja: Efecto de los extractos sobre las propiedades de oleogeles

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Received: January 10, 2024; Accepted: March 13, 2024

Abstract
This work aimed to compare the assisted extraction of polyphenol compounds from red cactus pear to elaborate stable oleogels, employing ultrasound or a high-shear impeller. A response surface method was employed to determine the simultaneous effect of temperature, soy lecithin percent, and ultrasound time or impeller speed, on polyphenols extraction employing soybean oil as solvent. The factors temperature, lecithin, and ultrasound time affected the amount of polyphenols extracted, although neither temperature nor lecithin presented an effect on polyphenols extraction in high-shear impeller extraction. With the enriched oil, two oleogels were elaborated using candelilla wax or soybean wax as oleogelators. Oleogels' firmness was higher in samples obtained with ultrasound treatment (10.05 and 4,85 for candelilla wax or soybean wax, respectively, and 7.80 and 1,10 for candelilla wax or soybean wax, respectively), but the use of the high-shear impeller decreased the peroxide value after 60 days. Candelilla wax oleogels were harder and more stable to oxidation than soybean wax oleogels. This means that the use of a high-shear impeller in polyphenols extraction is not temperature dependent, as in the case of ultrasound, enhancing extraction yield, resulting in more stable oleogels against lipid oxidation.
Keywords: ultrasound, high-shear impeller, polyphenols extraction, oleogels, oxidative stability.

Resumen
El objetivo de este trabajo fue comparar la extracción asistida de compuestos polifenólicos de tuna roja para elaborar oleogeles estables, empleando ultrasonido o un impulsor de alto corte. Se utilizó un diseño factorial para determinar el efecto simultáneo de la temperatura, porcentaje de lecitina de soya y tiempo de ultrasonido o velocidad del impulsor con aceite de soya como solvente. Los factores temperatura, lecitina y tiempo de sonicación afectaron la cantidad de fenoles extraídos, por otro lado, la extracción de polifenoles utilizando el impulsor de alto corte no fue afectada por la temperatura ni el contenido de lecitina. Con el aceite enriquecido se elaboraron dos oleogeles empleando cera de candelilla o cera de soya como oleogeladores. La firmeza de los oleogeles fue mayor en las muestras obtenidas con el tratamiento de ultrasonido (10.05 and 4,85 para cera de candelilla o cera de soya; respectivamente, y 7.80 and 1,10 para cera de candelilla o cera de soya, respectivamente), pero el uso del impulsor de alto corte disminuyó los valores de peróxido después de 60 días. Los oleogeles de cera de candelilla fueron más duros y estables a la oxidación que los oleogeles de cera de soya. Esto significa que el impulsor de alto corte en la extracción de polifenoles no depende de la temperatura, como en la extracción con ultrasonido, mejorando el rendimiento de extracción, y en oleogeles más estables a la oxidación de lípidos también.
Palabras clave: ultrasonido, impulsor de alto corte, extracción de polifenoles, oleogeles, estabilidad oxidative.

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https://doi.org/10.24275/rmiq/Alim24237
ISSN:1665-2738, *issn-e*: 2395-8472

1 Introduction

Agro-industrial co-products are secondary materials generated during the industrial processing of agricultural products, such as fruits or vegetables. These discarded materials can have a defined commercial market or not, and in the last case, these can generate an environmental problem, since waste represents the loss of raw resources and energy, implying investments to control pollution. These peels, rinds, and pomaces represent an important source of bioactive compounds (Silva *et al*., 2020). In general, phenolic compounds are the most abundant class of bioactive compounds with various and important biological functions and are secondary metabolites where the aromatic groups and hydroxyl groups content in their particular structure impart antioxidant properties in most cases (Sagar *et al*., 2018). Cactus pear peel is a source of important compounds as antioxidants (Cardador-Martínez *et al*., 2010) or prebiotics (Díaz-Vela *et al*., 2013).

The extraction of these biological compounds is commonly achieved by maceration, a solid-liquid extraction, one of the simplest techniques employed, where the temperature increases the kinetic energy of the molecules in the solid phase and the agitation allows the continuous diffusion of extracting solvent (Naviglio *et al*., 2023). Organic solvents with different polarities are employed to extract from the solid matrix the compounds of interest with different solubility, and the solvent is then evaporated by rotary evaporation (Giada, 2013). Solvents as water, absolute ethanol, ethanol:water, glycerol:water, or propylenglycol:water, and maceration time have a marked effect on total polyphenols extraction (García-Márquez *et al*., 2012).

Nonetheless, several techniques to improve the extraction yield have been proposed. For example, assisted extraction with ultrasound in the recovery of bioactive compounds from agro-industrial coproducts has advantages such as less time and energy consumption, besides a higher extraction yield with a lower quantity of solvents (Kumar *et al*., 2021). Quiroz-Reyes *et al*. (2913) reported that ultrasound assisted extraction resulted in higher antioxidant activity, related directly to total polyphenolic content, as compared to maceration extraction. This technique, as compared to Soxhlet, produced similar oil recovery with no change in the fatty acids composition, as well as resulted in similar recoveries of total polyphenols and tannins but with lower solvent consumption (Da Porto *et al*., 2013). Agitation is necessary during maceration to promote diffusion and recirculate the concentrated solvent from the cell material surface to the liquid phase, increasing extraction yield (Jha and Sit, 2022). Thus, mechanical agitation with conventional impeller or high-shear devices can be employed in this process. Xu *et al*., (2022) conducted extraction of polyphenol of walnut pellicle fibers by using mechanical agitation at different temperatures. Kwak *et al*., (2019) applied a high shear mixer to extract lipids of microalgae. High shear impellers also are characterized by the ability of produced high values of shear and are widely used for break-down and disperse particle cluster of pigments in a liquid vehicle (Guadarrama-Pérez *et al*., 2020). Nevertheless, to the best of the authors knowledge, high shear impellers has not been used to extract bioactive compounds.

The high shear enhances the solvent flow and can break down cell wall material to allow more solvent circulation. The dispersion of the solid material can be achieved by high-speed rotor-stator, and bioactive compounds can then be dispersed by co-dissolution, dispersion, or emulsification (Freitas *et al*., 2005). Although ultrasound is more capable than orbital agitation in recovering phenolic compounds, the use of impeller agitation at 60 °C can be a suitable replacement for polyphenol extraction (Xu *et al*., 2022). Oil temperature above 50 °C during extraction, irrespective of the assisted technique, is important because enhances oil fluidity decreasing viscosity, and favoring the extraction of bioactive compounds (Hlaváč *et al*., 2019).

Edible oils are non-volatile, economically viable, easily regenerated, and safe solvents due to their dissolving power (Li *et al*., 2014). In this view, and since edible oils are lipophilic non-polar systems with a wide variety in composition due to the source, quality, and extraction method, they can be employed as solvents in conjunction with assisted technologies such as ultrasound or microwaves (Yara-Varón *et al*., 2017). According to Li *et al*. (2017; 2019), the addition of endogenous amphiphilic compounds like partial glycerides and phospholipids, such as soy lecithin, increased the dissolving power of edible oils, improving the total polyphenols content extraction. Nonetheless, the edible oils' fatty acids composition is an indicator of nutritional properties but also influences its oxidative stability, since a relatively higher amount of unsaturated fatty acids with more double bonds are subjected to oxidization, decreasing oil stability (Choe and Min, 2006). In this case, the enriched oils with polyphenols extracted from agro-industrial coproducts will enhance their oxidative stability.

The use of oleogelators molecules, typically low-molecular weight species below 300 Da, promotes the self-assemble during cooling to form a thermos-reversible three-dimensional network that entraps the surrounding oil phase to form a gelled solid-like oleogel (Blake *et al*., 2018). Among the gelators that are able to form three-dimensional structures are

natural vegetable waxes, containing triacyl glycerides, long-chain esters, n-alkanes, and fatty acids, that crystalize to entrap liquid oil (Narine and Marangoni, 1999). These waxes possess the capacity to delay lipid oxidation, either the residual presence of phenolic compounds or the increase in viscosity that indirectly retard the rate of lipid oxidation (Valoppi *et al*., 2020).

The objective of this work was to compare the effect of two assisted solid-liquid extraction methodologies, ultrasound or high-shear impeller (Norstone impeller), on extracted polyphenols from red cactus pear peel, employing soybean oil as solvent plus soy lecithin, in order to formulate two different oleogels, employing candelilla wax or soybean wax as oleogelators, to evaluate their texture and oxidative stability.

2 Materials and methods

2.1 Raw materials and polyphenols determination

Red cactus pear (*Opuntia ficus-indica* L.) was collected at Jocotitlán, Estado de México, México, during the season from May to September 2022. Red cactus pear is an abundant wild fruit with no commercial value and hence, not consumed (Fig. 1). Fruits were recollected and transported to the laboratory, washed with tap water, and manually peeled. Peels were sun-dried and grounded in a grain mill before being sieved consecutively in No. 50 and 20 sieves to obtain a regular and homogeneous powder named flour, mixing different lots in a single batch (Chavez-Zepeda *et al*., 2009). Red cactus pear peel flour was stored in hermetic containers until use.

Red cactus pear (*Opuntia ficus-indica* L.) was collected at Jocotitlan, State of Mexico, Mexico, during the season from May to September 2022. Red cactus pear is an abundant wild fruit with no commercial value and hence, not freshly consumed (Fig. 1). Fruits were recollected and transported to the laboratory, washed with tap water, and manually peeled. Peels were sun-dried and grounded in a grain mill before being sieved consecutively in No. 50 and 20 sieves to obtain a regular and homogeneous powdered flour. Different lots were mixed into a single batch (Chavez-Zepeda *et al*., 2009). The red cactus pear peel flour was stored in hermetic containers until required for use. Soybean oil (Nutrioli®, RAGASA, Monterrey, Mexico) added with different soy lecithin (Kickapu, S.A. de C.V., Mexico) concentrations was used as solvent.

Oleo-extraction of polyphenols was adapted from Li *et al*. (2019). A solid (S; red cactus pear flour)-to-liquid (L; soybean oil w/added soy lecithin in different concentrations) ratio (SLR) of 1:4 (w/v) was used for this purpose.

Figure 1. Red cactus pear is a wild fruit with no commercial value and lower consumption at Jocotitlán, Estado de México, México.

The total polyphenols content in macerated oil was determined by modified liquid-liquid extraction by mixing 10 mL of oil sample with n-hexane (1:1, v/v) and then adding 20 mL of ethanol-water mixture (60:40, v/v) three times. Aqueous ethanolic extracts were combined and washed with n-hexane before filtering, and then evaporated to obtain a ca. 5 mL concentrated sample. Total polyphenol content was determined employing Folin-Ciocalteu reactive (Singleton and Rossi, 1965), adding one mL to one mL of sample, mixed with 8 mL of 0.7 M Na_2CO_3 for a final volume of 10 mL. The mixture was allowed to stand 2 h at room temperature in the dark and absorbance was measured at 765 nm and extrapolated against a catechol standard curve (0 to 100 mg/mL) to report mg of catechol equivalent per g of sample.

2.2 Assisted extraction methods

Two different assisted extraction methods were employed in the polyphenol substances extraction: ultrasound or high-shear impeller.

The ultrasound-assisted extraction was made adapting the methodology reported by Monzón *et al*., (2021), employing a Cole Parmer model 8892 ultrasonic cleaner (Cole Parmer, Vernon Hills, USA), at 80 W of power and a 40 kHz frequency. Sealed vessels with 80 g of soybean oil with 20 g of red pear peel flour were submerged into the tank during the time, lecithin percent, and temperature provided according to the experimental design (Table 1). Samples were let to settle for 24 h, and filtered before polyphenols content was determined.

The high-shear impeller extraction was made employing a Norstone® type impeller (ϕ= 49 mm, h= 6 mm, groove ϕ= 3 mm) fixed to the vertical axis of a Cole-Parmer digital mixer system model OS-200 (Cole-Parmer, Vernon Hills) placed at 28.8 mm from the bottom to accomplish the diameter/distance relationship of 0.3048 (Martínez-de Jesús *et al*., 2018). Samples of 160 g of soybean oil and 40 g of red pear peel flour, plus lecithin, were placed in cylindrical

Table 1. Three factors with three levels of factorial design generated by Design Expert® Software.

Run	Lecithin concentration (%)		Temperature (° C)		Ultrasound time or impeller speed		
	Coded	Uncoded	Coded	Uncoded	Coded	Uncoded (min)	Uncoded (rpm)
1	-1	0	-1	20	-1	30	800
2	1	2	-1	20	-1	30	800
3	-1	0	1	60	-1	30	800
4	1	2	1	60	-1	30	800
5	-1	0	-1	20	1	60	1,800
6	1	2	-1	20	1	60	1,800
7	-1	0	1	60	1	60	1,800
8	1	2	1	60	1	45	1,800
9	0	1	0	40	0	45	1,300
10	0	1	0	40	0	45	1,300

tanks without baffles (ϕ= 75 mm), in a temperature-controlled water bath. In all cases the residence time was 45 min. Table 1 shows the impeller speed-temperature combinations used for a given lecithin concentration. Samples were let to settle for 24 h, and filtered before polyphenols content determination was carried out.

2.3 *Polyphenols enriched oil structuration= oleogels firmness and oxidative stability*

Oleogels were formed by using either candelilla wax (3% w/v, Toro-Vazquez *et al*., 2007) or soybean wax (20% w/v, Sena *et al*., 2022) as oleogelators. The polyphenol-rich soybean oil/lecithin obtained by the ultrasound or high-shear impeller assisted extraction were heated were heated to 80 °C for 25 min in an oven, and then the waxes were added and mixed with magnetic stirring until they melted. Samples were poured into 20 mL beakers, covered with Saran®, and cooled down at room temperature overnight.

Oleogel texture was determined with a Brookfield LFRA Texture Analyzer (Brookfield Engineering Laboratories, Middleboro, USA). Samples were penetrated with a 10 mm diameter acrylic probe, at one mm/s, to an objective distance of 20 mm (ca. 30% of total oleogel height), reporting the sample firmness (in N) as the maximum force detected during penetration.

The protective antioxidant activity of the extracted polyphenols in the different oleogels was determined as the peroxide value according to the 965.33 AOAC Official Method (AOAC, 1999). Briefly, 5 g of oleogel was dissolved in 30 mL of acetic acid-chloroform solution (1:1, v/v), and then 0.5 mL of a fresh KI saturated solution was added and mixed for one minute, before adding 30 mL of distilled water. Samples were titrated with a 0.1 N sodium thiosulfate solution until the yellow coloration was gone, and then 0.5 mL of 0.1% starch solution freshly prepared was added to continue the titration until the blue coloration disappeared. Peroxide value was reported as O_2 milli-equivalents per kg of sample.

2.4 *Experimental design and data analysis*

A response surface methodology was employed to determine the simultaneous effect of three factors: soy lecithin concentration, as X1, in addition to temperature, X2 (°C) and, time (min, in the ultrasound-assisted extractions) or agitation speed (rpm, in the high-shear impeller-assisted extraction), X3 (%), on the oils' polyphenols content extracted by the two assisted extraction techniques (Table 1). Experimental data were analyzed in Design Expert® software to generate a linear equation according to:

$$Y = \beta_0 + \beta_1 X1 + \beta_2 X2 + \beta_3 X3 + \beta_{12} X1X3 + \beta_{13} X1X3 + \beta_{23} X2X3 + \beta_{123} X1X2X3 \quad (1)$$

Where Y is the extracted polyphenols, β_0 is the intercept, β_1, β_2, and β_3 are linear terms for lecithin concentration, temperature, and time or rpm, respectively, and β_{123} is the triple interaction of the factors employed. The significance of terms and cross-products and the adequacy of the model (R^2) were obtained from the ANOVA obtained in the same software.

The effect of the different assisted extraction methods (ultrasound or high-shear impeller) and the different oleogelator types (candelilla wax or soybean wax) on oleogel firmness and peroxide values was determined according to the model:

$$Y_{ij} = \mu + \alpha_i + \beta_j + \varepsilon \quad (2)$$

Where Y_{ij} represents the firmness or peroxide value of the oleogels for the i-th assisted extraction method (ultrasound or high shear impeller) and j-th oleogelator type (candelilla wax or soybean wax), where μ is the overall mean, α_i is the main effect of the type

Table 2. Results for the total polyphenols content with the two different assisted extraction methods: ultrasound or high-shear impeller.

Run	Coded variables			Total polyphenols content (mg of catechol equivalent/g of the sample)	
	Lecithin concentration (%)	Temperature (°C)	Ultrasound time (min) or impeller speed (rpm)	Ultrasound-assisted extraction	high-shear impeller-assisted extraction
1	-1	-1	-1	6.7657	16.8774
2	1	-1	-1	9.5846	9.6968
3	-1	1	-1	17.5051	12.7174
4	1	1	-1	10.7583	14.4677
5	-1	-1	1	7.7399	11.458
6	1	-1	1	14.1345	14.3197
7	-1	1	1	10.5975	9.8092
8	1	1	1	20.1634	19.6636
9	0	0	0	13.6659	16.1469
10	0	0	0	15.2229	14.2643

of assisted extraction, and β_j is the main effect of the oleogelator type; and ε is the error terms of a presumed normal distribution N (μ, σ^2) (Der and Everitt, 2001). Data were analyzed employing an analysis of variance using the R Studio® platform (https://www.rstudio.com/), and the significant difference (p <0.05) between means was determined by Tukey's honestly significant difference (HSD) in the same platform.

3 Results and discussion

The results of the total polyphenols extracted employing the two different assisted extraction methods are listed in Table 2. For the ultrasound-assisted method, the range of polyphenol-related substances was from 6.76 to 20.16 mg CE/g, and for the high-shear impeller-assisted method was from 9.69 to 19.66 mg CE/g. For both extraction methods, , the maximum amount of polyphenols extracted occurred at 2.0 % (w/v) lecithin concentration (2.0 %, w/v), 60 °C, and 60 min of ultrasound or an impeller speed of 1,800 rpm.

ANOVA results for the extracted polyphenols employing the ultrasound-assisted method are depicted in Table 3. The regression model presented a highly significant (P <0.01) effect. The linear parameters for time, temperature, and percent of soy lecithin and the cross-products terms presented as well a highly significant (P <0.01), and only the soy lecithin temperature interaction was non-significant (P >0.05). The fitting model presented a higher adjusted R^2 (0.9548). For the high-shear impeller-assisted extraction method (Table 3), the model presented a highly significant (P <0.01) effect. For the linear parameters, a highly significant effect (P <0.01) was only observed in the impeller speed. The temperature presented a significant (P <0.0) effect, with non-significant (P >0.05) effect due to soy lecithin percent. The three cross-products were highly significant (P <0.01), although the triple interaction was non-significant (P >0.05). The fitting model presented an acceptable adjusted R^2 (0.9113) value.

These results imply that for the ultrasound-assisted extraction, the time, temperature, and percent of soy lecithin influenced the total polyphenol substances extraction. In contrast, for the high-shear impeller-assisted extraction, speed and to a lesser degree temperature have an impact on polyphenols extraction. Both methods imply the use of mechanical force to enhance the extraction of the compounds of interest, where, on one hand, sonication results in a cavitation process that causes cells' swelling and/or cell walls' breakdown, allowing the increase of solvent diffusion rate across the cell walls that theoretically increase the washing out cell components (Kahan *et al*., 2010). On the other hand, when mechanical-assisted extraction is reported, high shear dispersers are employed to achieve the homogenization process of two immiscible systems, the solvent and plant material, to extract bioactive compounds from plant matrices (Procopet & Oroian, 2022). The sample is subjected to high shear force, homogenizing it to smaller particle size, since the rotational motion of the rotor creates a suction that draws the sample between the rotor and stator spaces (Kaur *et al*., 2004). This intense destruction of cellular structures as a result of the rotor's high speed generates shear, cavitation, and turbulence leading to the breakdown of plant material, and increasing the extraction of bioactive compounds as well (Gómez-Hoyos *et al*., 2022).

Table 3. ANOVA results for the different assisted oleo-extraction of polyphenols (ultrasound or high shear impeller) from red cactus pear peel.

Ultrasound-assisted extraction method					
Source	Sum of squares	DF	Mean square	F-value	p-value
Model	263.03	7	37.58	49.26	< 0.0001
A-Time	29.88	1	29.88	39.17	0.0001
B-Temperature	93.1	1	93.1	122.07	< 0.0001
C-Soy lecithin	10.21	1	10.21	13.38	0.0052
AB	15.96	1	15.96	20.93	0.0013
AC	84.36	1	84.36	110.6	< 0.0001
BC	0.1279	1	0.1279	0.1678	0.6917
ABC	29.4	1	29.4	38.54	0.0002
Std. Dev.	0.8733				
R^2	0.9746				
Mean	12.23				
Adjusted R^2	0.9548				
C.V. %	7.14				
Predicted R^2	0.8983				
High-shear impeller-assisted extraction method					
Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	146.98	7	21	24.48	<0.0001
A-Speed	11.44	1	11.44	13.33	0.0053
B-Temperature	7.59	1	7.59	8.84	0.0156
C- Soy lecithin	0.2755	1	0.2755	0.3212	0.5847
AB	43.36	1	43.36	50.55	<0.0001
AC	75.03	1	75.03	87.47	<0.0001
BC	9.29	1	9.29	10.83	<0.0001
ABC	0.0058	1	0.0058	0.0067	0.9365
Std. Dev.	0.9261				
R^2	0.9501				
Mean	13.85				
Adjusted R^2	0.9113				
C.V. %	6.69				
Predicted R^2	0.8004				

According to both contour graphs and regression equations, the effect of assisted-method extraction was more noticeable in high-shear impeller samples, with a higher intercept value (β_0= 13.68) as compared to ultrasound-assisted samples (β_0= 11.95). First order regression model involves two factors: the factor associated with linear effects, and the factor associated with the single cross-product, representing the linear interaction of the parameters. The coefficient parameters for the linear temperature and percent of soy lecithin terms were higher in the ultrasound-assisted extraction since these were less significant in the high-shear impeller-assisted extraction method. This is that ultrasound extraction depends on temperature and the presence of dissolving help agents such as lecithin to increase polyphenols extraction. However, most of the cross-product terms presented a significant effect on the total polyphenols extracted. In the ultrasound-assisted extraction (Fig. 2a, left) lower temperatures and times resulted in lower polyphenols extraction (parameter value= -0.99 time*T°C), but when a higher percent of soy lecithin was employed along with higher process time, the polyphenols extraction increased (parameter value= +2.30 time*%SL) (Fig 2a, center). Because the temperature and percent of soy lecithin interaction (Fig 2a, right) was not significant, the surface was flat, and the parameter value was the lower one (-0.09 T°C*%SL). For the high-shear impeller extraction, in the temperature and speed interaction, the temperature axis looks flat (no significant effect), and a more pronounced slope was present at the speed axis, with higher extracted polyphenols (parameter value= +1.65 T°C*rpm) (Fig. 2b, left). A similar pattern was observed in the percent of soy lecithin interaction with speed (parameter value= +2.17 %SL*rpm) (Fig. 2b, center).

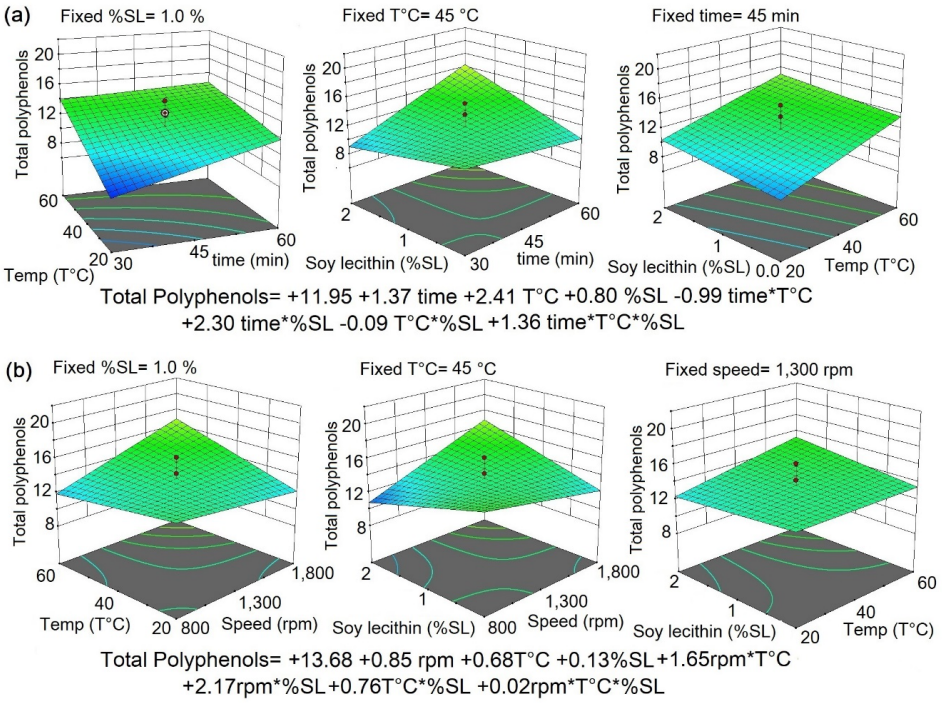

Figure 2. Regression surface graphs for the extracted polyphenols from red cactus pear employing (a) ultrasound-assisted extraction or (b) high-shear impeller-assisted extraction.

Table 4. Mean test for the oleogels formulated with the oil employed in the oleo-extraction of phenolic compounds by assisted extraction methods.

Assisted oleo-extraction method	Oleogel firmness (penetration force, N)		Peroxide value (mEq O2/kg) after 60 days of storage	
	Candelilla wax	Soybean wax	Candelilla wax	Soybean wax
Ultrasound	10.05±0.45 a, A	4.85±0.08 a, B	16.01±0.11 b, A	27.95±0.10 b, B
High shear impeller	7.85±0.11 b, A	1.10±0.10 b, B	11.89±0.45 a, A	29.80±0.08 a, B

a, b means with the same letter in the same column are not significantly different for the assisted extraction method. A, B means with the same letter in the same row are not significantly different for the oleogelator agent.

Finally, for the percent of soy lecithin interaction with temperature, the plotted surface was practically plane, and the parameter value was the second lower one (+0.76 %SL*T°C) (Fig. 2b, right).

For the oleogels elaborated with the two different oleogelators, the firmness of the oleogel samples was significant (P <0.05) higher for the ultrasound-assisted treatment. In the same manner, oleogels elaborated with candelilla wax presented significant (P <0.05) higher firmness values (Table 4) as well. These structural differences may be attributed, on one hand, to the inherent differences due to the nature and composition of the different waxes, and related aspects during wax processing, such as purity and/or presence of contaminants, resulting in different oleo-gelation capacity (Blake *et al*., 2018; Yilmaz *et al*., 2021). On the other hand, the textural differences determined as firmness (maximum force detected during compression) were related as well to the differences in crystal morphologies formed in the oleogel (Yilmaz *et al*., 2015). These inherent differences in the waxes' composition have a great influence on the oleogels' molecular organization, consequently affecting mechanical properties (Blake *et al*., 2018). As a consequence, the gelling concentration between candelilla wax and soybean wax is different, depending on the size and spatial distribution of the crystals formed during cooling down, being reflected in oleogels texture (Blake *et al*., 2014). Also, the added emulsifiers to improve polyphenols extraction, such as lecithin, can alter the structure and mechanical properties of the wax-based oleogels, enhancing elasticity and viscosity by modifying the crystalline structures affecting the lipid polymorphism during gelation.

The peroxide value, as related to lipid oxidation

and rancidity, was significant (P <0.05) higher for the ultrasound-assisted treatment. The significant (P <0.05) lower peroxide values were detected in the candelilla wax oleogels (Table 4). The stability of the oleogel depends on the properties and fatty acid composition of the oleogelator employed (Kupiec *et al*., 2020) since different waxes have different components more susceptible to oxidation or prooxidants components that during the oleogelation process were not able to avoid unsaturated fatty acids oxidation (Yilmaz *et al*., 2021). In this respect, Rohilla *et al*. (2023) reported that the increase in acid and peroxide values in edible oil with tamarillo peel as the source of carotenoids as antioxidants in high shear disperser extraction samples were related to higher mechanical shear, as compared to ultrasound-assisted extraction samples. Tissue disruption provoked by the high shear homogenizer results in endogenous lipids to fatty acids, and then oxidation (Boukobza *et al*., 2001). Norstone high-rate propeller did not provoke cell disruption, only a more efficient dispersion of the solvent throughout the vegetal matrix to extract polyphenol-related substances. In addition, the fatty acid composition related to and higher amounts of tocols and sterols present in oils could be a factor that promotes oxidative rancidity (Öğütcü and Yılmaz, 2015), so the incorporation of naturally extracted antioxidants from agro-industrial coproducts is a good alternative to improve their oxidative stability.

Conclusion

At the present experimental condition, the use of the Norstone high-shear impeller improved the oxidative stability of both oleogels, concomitantly with the higher extraction of polyphenol substances. Inherent textural differences were related to the type of wax employed, but the use of a high-shear impeller improved the polyphenol substances, showing no dependence on temperature. This means that this type of impeller can be employed as a good alternative to extract this kind of bioactive compounds, to improve the oxidative stability of oils, which can be further employed in culinary or industrial processes that imply lipid oxidation.

Acknowledgements

This research was supported into the proyect "Aprovechamiento de recursos agroindustriales subutilizados: Recuperación de compuestos antioxidantes de tuna roja utilizando aceite vegetal como solvente en la extracción asistida con ultrasonido o impulsores de alto corte, FICDTEM-2023-148", Fondo para la Investigación Científica y Desarrollo Tecnológico del Estado de México, COMECyT, Estado de México, México.

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