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Effect of different extraction methods on cottonseed oil yield

Efecto de diferentes métodos sobre el rendimiento de extracción de aceite de semilla de algodón

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Abstract

Cotton (*Gossypium hirsutum* L.) is an important fibre crop of global significance. It is mainly utilised for the textile industry, and its seeds as cattle feed and to re-harvest fields. However, an important amount of the cottonseed obtained during processing is discarded. Some reports indicate that cottonseed contains high-quality protein and important lipid content. The aim of the present study was to compare different extraction methods (Soxhlet, mechanical and ultrasound-assisted) in order to achieve the highest extraction yield of cottonseed oil (CSO). The extraction efficiency was measured based on the effects of temperature, organic solvent (OS), extraction time and solvent:seed (s:s) ratio, using a Box-Behnken 3^4 experimental design. From our results, ultrasound-assisted extraction had the highest extraction yield (38.25%) at 45 ºC, after 1 h for a 10:1 ratio using Folch mixture as OS. Palmitic, oleic and linoleic acids were the main residues in the characterised CSO, and this oil can be utilised for biodiesel production. This research intends to promote the use of this agroindustrial by-product to add value to cottonseed.

Keywords: cottonseed, oil, extraction, characterisation.

Resumen

El algodón (*Gossypium hirsutum* L.) es una fibra de importancia mundial. Se emplea principalmente para la industria textil, y sus semillas para alimentar ganado y para resiembra. Sin embargo, una cantidad importante de semilla de algodón es desperdiciada durante el procesamiento. Algunos estudios reportan que la semilla de algodón contiene proteínas de alta calidad y un contenido de lípidos considerable. Por ello, el objetivo del presente trabajo era comparar diferentes métodos de extracción (Soxhlet, mecánica y asistida por ultrasonido) para obtener el rendimiento más alto de aceite de semilla de algodón (CSO). La eficiencia de la extracción consideró los efectos de la temperatura, solvente (OS), tiempo de extracción, y relación solvente:semilla (s:s), bajo un diseño experimental Box-Behnken 3^4. Los resultados mostraron que con el método de ultrasonido se alcanza el rendimiento más alto (38.25%) a 45 ºC, 1 h, relación 10:1 y la mezcla de Folch como OS. Los ácidos grasos palmítico, oleico y linoleico se identificaron en el CSO caracterizado y el aceite se propone para preparar biodiesel. Esta investigación busca promover el uso de este residuo agroindustrial para revalorizar la semilla de algodón.

Palabras clave: semilla de algodón, aceite, extracción, caracterización.

1 Introduction

Cotton represents one of the most commercially important fibre crops attributed to its industrial and agricultural applications (Mendoza *et al*., 2016; Weiger *et al*., 2016; Egbuta *et al*., 2017; McCarty *et al*., 2018). Cotton is a semi-tropical or tropical climate crop; it is very sensitive: requires a properly conditioned land, deep soils, constant irrigation, and temperatures of 20 - 30 ºC to grow (SIAP, 2017). World cotton production during 2018/19 was 25.89×10^6 t (USDA, 2019), and it is expected to increase. Mexico was the ninth cotton-producing country during that period.

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According to data collected by SIAP (2017), from the 488 000 t of cotton produced in Mexico during 2016, 3.56% was reported as waste, cottonseeds included. Cottonseeds are the fifth most-produced oilseed worldwide, reaching 44.3 million t during the 2019/2020 oilseed production (www.statista.com). Okonkwo and Okafor (2016), mentioned that cottonseed is rich in high-quality protein and of remarkable lipid content (approximately 27.27 and 27.83%, respectively). Protein is commonly processed into premium cattle food (cottonseed meal, CSM) since it is rich in essential (16.66%) and non-essential (18.50%) amino acids (González-Vega and Stein, 2012). CSM is a by-product obtained from the extraction of cottonseed oil (CSO), which is another product of great relevance. The fatty acid (FA) profile of CSO comprises linoleic (55.38%), oleic (14.53%), and palmitic (27.39%) acids residues (Okonkwo and Okafor, 2016). Because of its FA content, CSO has been cited as suitable for shortenings, margarines, salad oil and biofuels (Dowd, 2011). Furthermore, CSO contains bioactive compounds such as antioxidants, including tocopherols, sterols, and flavonoids (Mariod and Mattäus, 2011; Nix *et al*., 2017), as well as pigments (gossypol; Tian *et al*., 2016), which can be useful for the enrichment, fortification of flavour and stabilization of shelf life for food and medical applications. The recovery of lipids from cottonseed has been carried out by conventional Soxhlet extraction (SE) (Conkerton *et al*., 1995; Saxena *et al*., 2011), which provides high yield but can lead to FA degradation. Non-conventional methods including supercritical fluid extraction (SFE) (Bhattacharjee *et al*., 2007) and microwave-assisted extraction (MAE) (Taghvaei *et al*., 2014, 2015) have also been applied. Nevertheless, SFE is costly and MAE requires a better understanding of the methodology to obtain reliable products (Samaram *et al*., 2015). A typical extraction procedure is conducted by mechanical means (ME), where no organic solvents are used but lower extraction yields are attained (De Conto *et al*., 2011). Ultrasound-assisted extraction (UAE), which is another alternative, has not been employed for CSO extraction, but for the extraction of oil from tobacco (Stanisaljveić *et al*., 2007), canola (Jalili *et al*., 2017), and soybean (Yousuf *et al*., 2018). UAE greatly enhances oil extraction without compromising the integrity of components, being a simple and affordable method (Samaram *et al*., 2013; Tian *et al*., 2013). It is worth noting that different yields are achieved for those extraction methods caused by parameters assessed, namely, temperature, extraction time, power, m/V ratio, pressure, particle size, and solvent flow rate, among others (Mwaurah *et al*., 2019). Therefore, the aim of the present research was to evaluate the efficiency of CSO extraction by means of SE, ME and UAE, as well as to investigate the effect of temperature, organic solvent (OS), extraction time and solvent:seed (s:s) ratio. The FA profile was also characterised. Our research intends to promote the use of this agroindustrial residue to add value to by-products from cotton processing.

2 Materials and methods

2.1 Plant material and reagents

Cotton (*Gossypyum hirsutum* L.) was cultivated and harvested in the local area of Delicias, Chihuahua, Mexico, during April - October 2017, where it is processed for the textile industry. Cottonseeds were obtained from the "Despepitadora Lázaro Cárdenas", which is located at the following coordinates: 28.370451, -105.603925. The fatty acid standard Supelco 37 Component FAME Mix was purchased from Supelco (Bellefonte, PA). All the solvents employed were of reagent grade from J.T. Baker (New Jersey, USA).

2.2 Lipid content

Collected cottonseeds were cleaned manually and then subjected to a thermal bath treatment (5 min, 60 ºC) for easier removal of the hull. After that, cottonseeds were dried overnight at 60 ºC in an incubator (Felisa model FE-133D; Felisa®, Jalisco, MX). Cottonseeds were then ground for oil extraction with an IKA all basic mill (IKAWorks, Inc., Wilmington, NC). Three different extraction methodologies were employed: Soxhlet extraction (SE), ultrasonic assisted extraction (UAE) and mechanical oil extraction (ME). Extraction process variables were: temperature (30, 45 and 60 ºC), organic solvent (n-hexane, Folch, $CHCl_3$-CH_3OH [1:2]), extraction time (20, 40, and 60 min) and solvent:seed ratio (10:0.5, 10:1.0 and 10:1.5 mL/g). For SE, seeds were placed in the corresponding apparatus and combined with the different organic solvents (OS) while magnetically stirred. Regarding UAE, amber vials containing the sample and the different OS were set in the equipment (Sonicator VWR model 150 D; VWR International., West Chester, PA). The resulting mixtures obtained

from both procedures were filtered through filter paper #1 (125 mm ø; Whatman, Maidstone, UK) and then heated at 60 ºC in a hot plate to remove the OS. Lipid content obtained in the different extracts was estimated by weight difference. In the case of ME, dry peeled cottonseeds were pressed in a manual expeller machine. For the three methods, the extraction yield (EY) was calculated with Equation 1:

$$\text{Extraction yield (\%)} = \frac{\text{oil weight}}{\text{seeds weight}} \times 100 \quad (1)$$

2.3 *Fatty acid profile*

The FA profile of CSO was determined by gas chromatography. First, the CSO obtained was subjected to selective derivatization following the protocol reported by Miranda *et al*. (2013): 200 μL of CSO were mixed with 1 mL of 0.2N HCl-CH_3OH and then heated at 60 °C during 4 h; then, 0.2 mL of distilled water and 2 mL of hexane were added. Secondly, two μL of the extract were injected into a HP Model 6890 gas chromatograph fitted with a flame ionization detector (FID) and a HP-INNOWAX polar capillary column (30 m X 0.32 mm X 0.25 μm). The temperature program was according to López-García *et al*. (2017). FA were identified by comparing their retention times with those observed in a Supelco 37 FAME Mix standard.

2.4 *Experimental design and statistical analysis*

A set of 27 treatments in a Box-Behnken 3^4 experimental design was employed to determine the best extraction conditions to reach the highest extraction yield (response variable). The factors were: temperature (30, 45 and 60 ºC), organic solvent (n-hexane, Folch, $CHCl_3$-CH_3OH [1:2]), extraction time (20, 40, and 60 min) and solvent:seed ratio (10:0.5, 10:1.0 and 10:1.5 mL/g). All the experiments were carried out by triplicate and a quadratic model (Equation 2) for the prediction of the dependent variable was used:

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i<j} \sum \beta_{ij} x_i x_j + \varepsilon \quad (2)$$

where y is the response variable (extraction yield, EY), β_0, β_i, β_{ii} and β_{ij} represent the regression coefficients of the combined, lineal, quadratic and interaction effects, respectively; x_i and x_j are the independent variables. To fit the second-order model, experimental data was analysed statistically using MINITAB®18 software (Minitab Inc., State College, PA). Differences among treatment mean values were evaluated through analysis of variance (ANOVA) and Tukey test using the SAS university edition software (SAS Institute Inc., Cary, NC), and statistical significance was set at $\alpha = 0.05$.

3 Results and discussion

Due to the cultural, economic and biological relevance of cotton, the present study deals with the extraction and characterization of the lipid content of one of its by-products: cottonseeds. For that purpose, three different methodologies were evaluated in an effort to recover the maximal amount of CSO.

3.1 *Lipid content*

As a first approach, we followed the Mexican official method NMX-F-089-S-1978 to determine the lipid content. The amount of lipids of the cottonseed collected was 28.68%. After that, in an effort to improve the extraction yield, SE, ME and UAE methods were evaluated and the results obtained are depicted in Table 1. As it can be observed, UAE allowed to reach the highest EY: 38.25%.

Table 1. Oil extraction yields from cottonseed by three different methods.

Extraction method	OS	EY (%)
SE	n-hexane	23.18 ± 1.95^{d}
	Folch*	20.19 ± 1.42^{e}
	$CHCl_3$:CH_3OH (1:2)	28.18 ± 2.18^{cd}
ME	-	9.79 ± 0.71^{f}
UAE	n-hexane	23.63 ± 1.81^{d}
	Folch*	38.25 ± 0.52^{a}
	$CHCl_3$:CH_3OH (1:2)	30.04 ± 2.60^{bc}

SE: Soxhlet extraction, ME: mechanical oil extraction, UAE: ultrasonic assisted extraction; OS: organic solvents; EY: extraction yield. Data are means ± standard error (n = 6). Different superscript letter in the same column indicate significant difference ($p < 0.05$). * Folch = $CHCl_3$:CH_3OH (2:1).

Table 2. Experimental setup for the four-factor, three-level Box-Behnken design and the response yield after analysis.

Experiment	x_1	x_2	x_3	x_4	y_1
1	30	Folch	40	10:01.0	29.17 ± 2.08^{c}
2	60	Folch	40	10:01.0	30.01 ± 1.41^{bc}
3	30	$CHCl_3$-CH_3OH (1:2)	40	10:01.0	21.80 ± 0.84^{fgh}
4	60	$CHCl_3$-CH_3OH (1:2)	40	10:01.0	21.68 ± 1.49^{fgh}
5	45	n-hexane	20	10:00.5	17.22 ± 1.20^{ghij}
6	45	n-hexane	60	10:00.5	23.63 ± 1.81^{def}
7	45	n-hexane	20	10:01.5	14.23 ± 0.56^{j}
8	45	n-hexane	60	10:01.5	17.94 ± 1.32^{ghij}
9	45	n-hexane	40	10:01.0	18.76 ± 0.87^{ghij}
10	30	n-hexane	40	10:00.5	22.11 ± 2.37^{efgh}
11	60	n-hexane	40	10:00.5	21.76 ± 2.14^{fgh}
12	30	n-hexane	40	10:01.5	16.46 ± 0.48^{ij}
13	60	n-hexane	40	10:01.5	17.20 ± 0.71^{hij}
14	45	Folch	20	10:01.0	27.19 ± 1.88^{cde}
15	45	$CHCl_3$-CH_3OH (1:2)	20	10:01.0	22.3 ± 1.14^{efg}
16	45	Folch	60	10:01.0	38.25 ± 0.52^{a}
17	45	n-hexane	60	10:01.0	21.81 ± 0.50^{fgh}
18	45	n-hexane	40	10:01.0	19.12 ± 1.72^{fghij}
19	30	n-hexane	20	10:01.0	17.84 ± 1.18^{ghij}
20	60	n-hexane	20	10:01.0	8.72 ± 1.03^{k}
21	30	n-hexane	60	10:01.0	21.60 ± 2.35^{fgh}
22	60	n-hexane	60	10:01.0	20.11 ± 2.27^{fghi}
23	45	Folch	40	10:00.5	34.63 ± 1.00^{ab}
24	45	$CHCl_3$-CH_3OH (1:2)	40	10:00.5	30.04 ± 2.60^{bc}
25	45	Folch	40	10:01.5	29.38 ± 1.64^{c}
26	45	$CHCl_3$-CH_3OH (1:2)	40	10:01.5	17.13 ± 0.46^{hij}
27	45	n-hexane	40	10:01.0	19.65 ± 2.00^{fghi}

X_1: temperature (ºC), X_2: organic solvent, X_3: extraction time (min), X_4: solvent:seed ratio (mL/g); y_1: extraction yield (%).

Such value was not only larger among UAE experiments but also greater than the other values achieved for the other methods: with respect to ME, UAE was 4-fold higher and compared to SE it was 26.33% higher. These results clearly indicate a significant effect of the extraction process variables. Table 2 summarizes the experimental setup where it can be seen that such variables led to different EY. The first entries that are important to highlight are experiments 16 and 20. The extraction conditions assayed in experiment 16 allowed to attain the highest EY, whereas the lowest EY (8.72%) was obtained under conditions of experiment 20. Secondly, we can refer the effect of OS, for instance, in experiment 17, the extraction process variables are the same except for the use of n-hexane instead of Folch's solvent mixture. In the case of experiments 1 and 2, OS allowed to attain a very similar EY; however, it can be mentioned that both temperature and extraction time affected EY, when experiments 1 and 2 are compared to experiment 16. On the other hand, for experiments 14, 23 and 25, the affecting variables were extraction time and solvent:seed ratio. The variations among these results can be attributed to their differences in polarities and viscosities (Tian *et al*., 2013). It is worth mentioning that the Folch mixture was the best OS for the recovery of CSO, followed by $CHCl_3$-CH_3OH (1:2), and n-hexane. The $CHCl_3$-CH_3OH mixture may interact with amphiphilic or polar components like phospholipids, improving the extraction yield (Kozłowska *et al*., 2016).

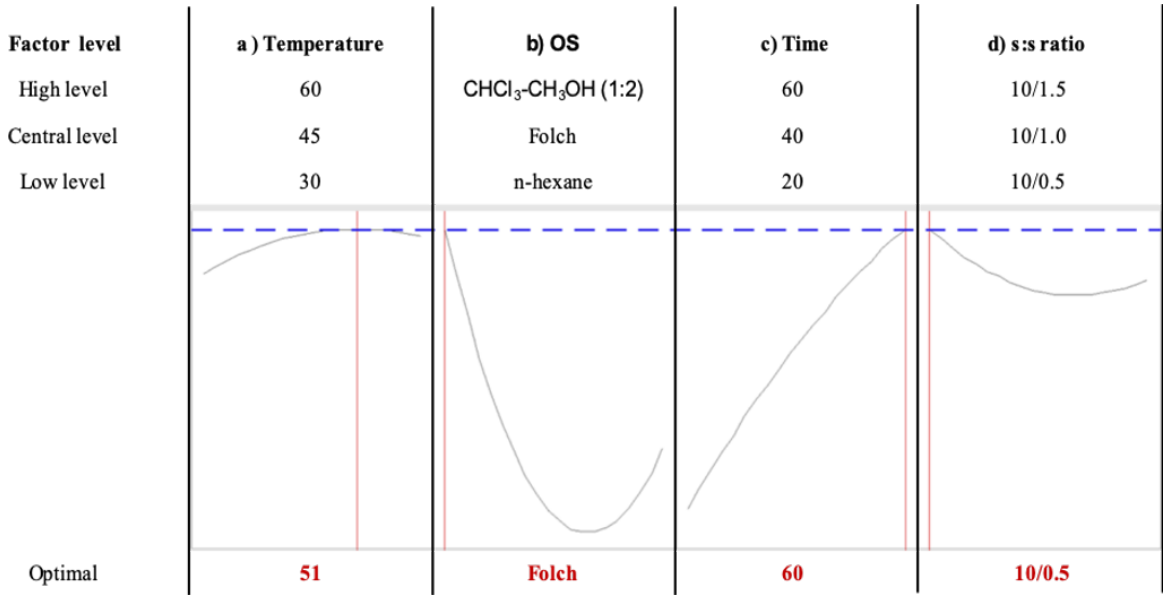
Finally, another remarkable aspect is that lower ratios lead to higher EY. This can be noticed in experiments 24 and 26, where for a 0.5 ratio EY

was almost twice, which is explained by the fact that larger amounts of ground cottonseed require a larger volume of OS. A similar behaviour can be observed for experiments 6 and 8; however, it cannot be said the same for experiments 23 and 25, where OS is clearly exerting a positive effect on EY.

3.2 Optimisation of the extraction process

The effects of the independent variables were described above and are shown in Figure 1. According to these data, an increase in temperature (Figure 1a) seems to favour the achievement of higher EY; however, in our findings we noted that 45 ºC was better than 60 ºC. Similar observations were found in the technical literature. Wong *et al*. (2019), reported an increase in hazelnut oil extraction when temperature was raised from 28 to 38 ºC, but from 38 to 48 ºC the oil yield extraction decreased. According to our optimisation, 51 ºC was the best temperature for the extraction of CSO, which is a slightly higher value than 45 ºC, the temperature for experiment 16. It is possible that at higher temperatures, the bubbles created by the cavitation forces collapsed with less intensity or more easily, decreasing their disrupting effect on the sample matrix; either because of an increase in the vapor pressure, developing a similar pressure between the inside and outside of the bubble, or by a decrease in the surface tension, affecting bubbles formation (Goula, 2013). Some reports indicate that higher temperatures lead to larger amounts of extracted oil. Samaram *et al*. (2015), mentioned that higher oil yields were reached from papaya seeds and hazelnuts, when temperature increased from 20 to 62.5 ºC. Zhang *et al*. (2009), cited that increasing temperature and extraction time, improved their oil recovery from almond powder by UAE, obtaining 81.89% of oil after 55 min and 51 ºC. On the other hand, Goula (2013) and Li *et al*. (2015), refer in their studies a small reduction in extraction yield, when temperature exceed 20 and 40 ºC, respectively. With respect to OS, Folch was a good solvent system for the recovery of CSO (Experiment 16, Figure 1b). The use of Folch as OS led to the most elevated EY, which is attributed to the polarities and viscosities of the solvents in the mixture.

Regarding to extraction time, longer times are supposed to lead to higher EY (Figure 1c). From our results, it can be observed that after 60 min the larger EY was reached as it is demonstrated by the optimisation. This is in agreement with Wong *et al*. (2019), who optimised the extraction of hazelnut oil via UAE, using ethanol as OS for 90 min (38 ºC and 90% of ultrasound amplitude) with an oil yield of 79.88%, an amount three-fold larger than the lowest yield reported, that corresponded to 30 min (28 ºC and 60% of ultrasound amplitude). This can be explained by the fact that longer times allow the oil contained in the sample to diffuse into the OS (de Mello *et al*., 2017).



a) ºC; b) organic solvent; c) min; d) mL/g

Fig. 1. Optimized graphics of the independent variables.

All three extraction process variables described before are affected by the solvent:seed ratio (Figure 1d). Chaniti and Tzia (2017) reported that increasing the ratio also increased their oil extraction yields. Employing high amounts of OS for low mass samples intensifies the solvent capacity to extract the oil from the sample matrix, since it produces a greater diffusion and therefore, a higher concentration gradient inside the sample, improving the transfer of the oil molecules to the liquid media (Samaram *et al*., 2015). The latter was observed in our results, when lower ratios led to higher EY. In fact, when the process was optimised, the 10:0.5 ratio was found to be the best.

According to the optimisation (see Table 3), the linear and quadratic terms of the OS (X_2 and $X22$, respectively), and the linear term of solvent:seed ratio (X_4) exerted the major effects on the extraction yield ($p < 0.001$), followed by the linear term of extraction time (X_3) ($p < 0.005$). On the other hand, the interaction terms of OS and extraction time (X_2X_3), as well as the one corresponding to OS and solvent:seed ratio (X_2X_4) were statistically significant ($p < 0.05$). By cancelling the non-significant factors, the mathematical model obtained expressed in terms of EY (y_1) is represented by Equation 3:

$$y_1 = 17.99 - 4.278X_2 + 9.150X_2^2 + 2.511X_3 - 3.646X_4 - 3.620X_2X_3 - 2.43X_2X_4 \quad (3)$$

where X_2 is OS, X_3 is extraction time, and X_4 is the solvent:seed ratio. The total regression coefficient (R^2) was 0.9556, which implies a good fitting since 95.56% of the total variability of the response variable could be explained by the mathematical model. From Equation (3), the optimal conditions for the oil extraction from cottonseed were: 51 ºC, Folch as OS, 60 min, and a solvent:seed ratio of 10:0.5; with a theoretical EY of 39.97%.

The predicted conditions were explored and an EY of 37.39 ± 0.73% was obtained. Such value was very close to the EY reached under experiment 16 (38.25 ± 0.52%). The different values for both theoretical EY and experimental one, are due to differences in temperature (51 and 45 ºC, respectively) as well as in ratio (10:0.5 and 10:1, respectively).

Table 3. Regression coefficients and *P* values of factors for the second-order model.

Factor	Coefficient	P value
Intercept	17.99	0
X_1	-0.943	0.152
X_2	-4.278	0
X_3	2.511	0.002
X_4	-3.646	0
$X_1 \cdot X_1$	-1.128	0.246
$X_2 \cdot X_2$	9.15	0
$X_3 \cdot X_3$	-1.199	0.219
$X_4 \cdot X_4$	1.898	0.062
$X_1 \cdot X_2$	-0.51	0.641
$X_1 \cdot X_3$	1.91	0.098
$X_1 \cdot X_4$	0.53	0.625
$X_2 \cdot X_3$	-3.62	0.005
$X_2 \cdot X_4$	-2.43	0.042
$X_3 \cdot X_4$	-0.32	0.771

X_1: temperature (ºC), X_2: organic solvent, X_3: extraction time (min), X_4: solvent:seed ratio (mL/g).

3.3 Fatty acid profile

Once the oil extraction was optimised, we proceeded to characterize the FA profile of the CSO (see Table 4). The main FA residues identified are: C18:2 (55.91%), C16:0 (20.91%) and C18:1 (16.54%). This profile agrees with the ranges mentioned by Thompson *et al*. (2019), for the same residues: 50-60%, 22-26% and 16-20%, respectively. The FA profile of the CSO analysed is constituted by 72.45% of unsaturated fatty acids (UFA), whereas the saturated fatty acids (SFA) amounted 24.96%, which means that the SFA/UFA ratio is 0.4. Mohdaly *et al*. (2017), reported a similar profile than that listed in Table 4 but included elaidic acid (2.10%) in their results. This difference can be attributed to the cotton species used, as well as agronomical procedures and geographical location. Nevertheless, their study also presented a comparable SFA/UFA ratio of 0.35, indicating both CSO are mainly composed by UFA.

It is worth mentioning that the FA profile obtained for CSO is of a remarkable value, for both nutritional and energy applications, including frying (Arslan *et al*., 2016), edible oleogels (Pehlivanoglu *et al*., 2018), and shortenings (Imran and Nadeem, 2015), among others products. In the context of energy applications, biodiesel production is a promising use (Jamshaid *et al*., 2018). With respect to biodiesel, the CSO obtained in the present study, it will be our raw material for transesterification reactions in a new and novel enzymatic process. As it was mentioned before, we intend to promote the use of this agroindustrial by-product to add value to cottonseed, as it is doing for other subtilized Mexican plants such as xoconostle

Table 4. Fatty acid profile of cottonseed oil.

Fatty acid	% wt.
C8:0	0.74
C10:0	2.43
C14:0	0.88
C16:0	20.91
C18:0	2.59
C18:1	16.54
C18:2	55.91
Saturated fatty acids	24.96
Unsaturated fatty acids	72.45
SFA/UFA ratio*	0.4

* Saturated/unsaturated fatty acid ratio.

(Dávila-Hernández *et al*., 2019) and calabacilla loca (Hernández-Centeno *et al*., 2020).

Conclusions

It was demonstrated that an agroindustrial by-product, cottonseeds, has potential nutritional and energy applications due to its lipid content. Different methods were evaluated for the extraction of CSO and UAE was found to be the best protocol for the recovery of CSO. When the extraction was optimised, different values for EY were reached for the theoretical and experimental values, indicating that the different extraction process variables exert a particular and combined effect on the overall process.

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Nomenclature

CSM	cottonseed meal
CSO	cottonseed oil
EY	extraction yield
FA	fatty acid
MAE	microwave-assisted extraction
OS	organic solvent
SE	Soxhlet extraction
SFA	saturated fatty acids
SFE	supercritical fluid extraction
SIAP	Servicio de Información Agroalimentaria y Pesquera
UAE	ultrasound-assisted extraction
UFA	unsaturated fatty acids
USDA	Departamento de Agricultura de los Estados Unidos

References

Arslan, F.N., Şapçi, A.N., Duru, F. and Kara, H. (2016). A study on monitoring of frying performance and oxidative stability of cottonseed and palm oil blends in comparison with original oils. International *Journal of Food Properties 20*, 704-717. https://doi.org/10.1080/10942912.2016.1177544

Bhattacharjee, P., Singhal, R.S. and Tiwari, S.R. (2007). Supercritical carbon dioxide extraction of cottonseed oil. *Journal of Food Engineering 79*, 892-898. https://doi.org/10.1016/j.jfoodeng.2006.03.009

Chanioti, S. and Tzia, C. (2017). Optimization of ultrasound-assisted extraction of oil from olive pomace using response surface technology: Oil recovery, unsaponifiable matter, total phenol content and antioxidant activity. *LWT - Food Science and Technology 79*, 178-189. https://doi.org/10.1016/j.lwt.2017.01.029

Conkerton, E.J., Wan, P.J. and Richard, O.A. (1995). Hexane and heptane as extraction solvents for cottonseed: A laboratory-scale study. *Journal of the American Oil Chemists' Society 72*, 963-965. https://doi.org/10.1007/bf02542075

Dávila-Hernández, G., Sánchez-Pardo, M.E., Gutiérrez-López, G.F., Necoechea-Mondragon, H. and Ortiz-Moreno, A. (2019). Effect of microwave pretreatment on bioactive compounds extraction from xoconostle (*Opuntia joconostle*) by-products. *Revista Mexicana de Ingeniería Química 18*, 191-204. https://doi.org/10.24275/uam/izt/dcbi/revmexingquim/2019v18n1/Davila

De Conto, L.C., Gragnani, M.A.L., Maus, D., Ambiel, H.C.I., Chiu, M.C., Grimaldi, R. and Gonçalves, L.A.G. (2011). Characterization of

crude watermelon seed oil by two different extractions methods. *Journal of the American Oil Chemists' Society 88*, 1709-1714. https://doi.org/10.1007/s11746-011-1850-8

de Mello, B.T.F., dos Santos Garcia, V.A. and da Silva, C. (2017). Ultrasound-assisted extraction of oil from chia (*Salvia hispânica* L.) seeds: Optimization extraction and fatty acid profile. *Journal of Food Process Engineering 40*, 1-8. https://doi.org/10.1111/jfpe.12298

Dowd, M.K. (2011). Cottonseed oil. In: *Vegetable Oils in Food Technology: Composition, Properties and Uses, Second Edition*, (F.D. Gunstone Ed.), Pp. 199-224. Blackwell Publishing Ltd.

Egbuta, M., McIntosh, S., Waters, D., Vancov, T. and Liu, L. (2017). Biological Importance of cotton by-products relative to chemical constituents of the cotton plant. *Molecules 22*, 93. https://doi.org/10.3390/molecules22010093

Gonzalez-Vega, J.C. and Stein, H.H. (2012). Amino acids digestibility in canola, cottonseed, and sunflower products fed to finishing pigs. *Journal of Animal Science 90*, 4391-4400. https://doi.org/10.2527/jas.2011-4631

Goula, A. (2013). Ultrasound-assisted extraction of pomegranate seed oil - Kinetic modeling. *Journal of Food Engineering 117*, 492-498. https://doi.org/10.1016/j.jfoodeng.2012.10.009

Hernández-Centeno, F., Hernández-González, M., López-De la Peña, H.Y., López-Trujillo, R., Zamudio-Flores, P.B., Ochoa-Reyes, E., Tirado-Gallegos, J.M. and Martínez-Vázquez, D.G. (2020). Changes in oxidative stability, composition and physical characteristics of oil from a non-conventional source before and after processing. *Revista Mexicana de Ingeniería Química 19*, 1389-1400. https://doi.org/10.24275/rmiq/Alim937

Imran, M. and Nadeem, M. (2015). Triacylglycerol composition, physico-chemical characteristics and oxidative stability of interesterified canola oil and fully hydrogenated cottonseed oil blends. Lipids in Health and Disease 14, 138. https://doi.org/10.1186/s12944-015-0140-0

Jalili, F., Jafari, S.M., Emam-Djomeh, Z., Malekjani, N. and Farzaneh, V. (2017). Optimization of ultrasound-assisted extraction of oil from canola seeds with the use of response surface methodology. *Food Analytical Methods 11*, 598-612. https://doi.org/10.1007/s12161-017-1030-z

Jamshaid, M., Masjuki, H.H., Kalam, M.A., Zulkifli, N.W.M., Arslan, A., Alwi, A., Khuong, L.S., Alabdulkarem, A. and Syahir, A.Z. (2018). Production optimization and tribological characteristics of cottonseed oil methyl ester. *Journal of Cleaner Production 209*, 62-73. https://doi.org/10.1016/j.jclepro.2018.10.126

Kozłowska, M., Gruczyńska, E., Ścibisz, I. and Rudzińska, M. (2016). Fatty acids and sterols composition, and antioxidant activity of oils extracted from plant seeds. *Food Chemistry 213*, 450-456. https://doi.org/10.1016/j.foodchem.2016.06.102

Li, H.Z., Zhang, Z.J., Hou, T.Y., Li, X.J. and Chen, T. (2015). Optimization of ultrasound-assisted hexane extraction of perilla oil using response surface methodology. *Industrial Crops and Products 76*, 18-24. https://doi.org/10.1016/j.indcrop.2015.06.021

López-García, G., López-Martínez, L.X., Dublán-García, O. and Baeza-Jiménez, R. (2017). Extraction and characterization of the fatty acid profile of quintonil (*Amaranthus hybridus*). *Revista Mexicana de Ingeniería Química 16*, 835-844. http://rmiq.org/ojs311/index.php/rmiq/article/view/953

Mariod, A. and Mattähus, B. (2011). Fatty acids, tocopherols, sterols of Cephaloctron cordofanus in comparison with sesame, cotton, and groundnut oils. *Journal of the American Oil Chemists' Society 88*, 1297-1303. https://doi.org/10.1007/s11746-011-1796-x

McCarty, J.C., Deng, D.D., Jenkins, J.N. and Geng, L. (2018). Genetic diversity of day-neutral converted landrace *Gossypium hirsutum* L. accessions. *Euphytica 214*, 173. https://doi.org/10.1007/s10681-018-2264-6

Mendoza, C.P., Gómez, M.R.T., González, Q.O., Padilla, F. and Corral, J.A.R. (2016). Genetic

resources of cotton in Mexico: ex situ and in situ conservation and use. *Revista Mexicana de Ciencias Agrícolas 7*, 5-16.

Miranda, K., Baeza-Jiménez, R., Noriega-Rodríguez, J.A., Otero, C. and García, H.S. (2013). Optimization of structured diacylglycerols production containing n-3 via enzyme-catalysed glycerolysis of fish oil. *European Food Research and Technology 236*, 435-440. https://doi.10.1007/s00217-012-1889-2.

Mohdaly, A., Seliem, K., El-Hassan, A. and Mahmoud, A. (2017). Effect of refining process on the quality characteristics of soybean and cotton seed oils. *International Journal of Current Microbiology and Applied Sciences 6*, 207-222. https://doi.org/10.20546/ijcmas.2017.601.026

Mwaurah, P.W., Kumar, S., Kumar, N., Attkan, A.K., Panghal, A., Singh, V.K., and Garg, M.K. (2019). Novel oil extraction technologies: Process conditions, quality parameters, and optimization. *Comprehensive Reviews in Food Science and Food Safety 19*, 3-20. https://doi.org/10.1111/1541-4337.12507

Nix, A., Paull, C. and Colgrave, M. (2017). Flavonoid profile of the cotton plant, *Gossypium hirsitum*: A review. *Plants 6*, E43. https://doi.org/10.3390/plants6040043

Okonkwo, S.I. and Okafor, E.C. (2016). Determination of the proximate composition, physicochemical analysis and characterization of fatty acid on the seed and oil of *Gossypium hirsutum. International Journal of Chemistry 8*, 57-61. https://doi.org/10.5539/ijc.v8n3p57

Pehlivanoglu, H., Ozulku, G., Yildirim, R.M., Demirci, M., Toker, O.S. and Sagdic, O. (2018). Investigating the usage of unsaturated fatty acid-rich and low-calorie oleogels as a shortening mimetic in cake. *Journal of Food Processing and Preservation 42*, e13621. https://doi.org/10.1111/jfpp.13621

Samaram, S., Mirhosseini, H., Tan, C.P. and Ghazali, H.M. (2013). Ultrasound-assisted extraction (UAE) and solvent extraction of papaya seed oil: Yield, fatty acid composition and triacylglycerol profile. *Molecules 18*, 12474-12487. https://doi.org/10.3390/molecules181012474

Samaram, S., Mirhosseini, H., Tan, C.P., Ghazali, H.M., Bordbar, S. and Serjouie, A. (2015). Optimisation of ultrasound-assisted extraction of oil from papaya seed by response surface methodology: Oil recovery, radical scavenging antioxidant activity, and oxidation stability. *Food Chemistry 172*, 7-17. https://doi.org/10.1016/j.foodchem.2014.08.068

Saxena, D.K., Sharma, S.K. and Sambi, S.S. (2011). Comparative extraction of cottonseed oil by n-hexane and ethanol. *International Journal of Engineering and Applied Sciences 6*, 84-89.

SIAP (2017). https://www.gob.mx/cms/uploads/attachment/file/257068/Potencial-Algod_n.pdf

Stanisavljević, I.T., Lazić, M.L. and Veljković, V.B. (2007). Ultrasonic extraction of oil from tobacco (*Nicotiana tabacum* L.) seeds. *Ultrasonics Sonochemistry 14*, 646-652. https://doi.org/10.1016/j.ultsonch.2006.10.003

Statista 2020. Worldwide oilseed production in 2019/2020, by type. Available at: https://www.statista.com/statistics/267271/worldwide-oilseed-production-since-2008/. Accessed: May 11th, 2020.

Taghvaei, M., Jafari, S.M., Assadpoor, E., Nowrouzieh, S. and Alishah, O. (2014). Optimization of microwave-assisted extraction of cottonseed oil and evaluation of its oxidative stability and physicochemical properties. *Food Chemistry 160*, 90-97. https://doi.org/10.1016/j.foodchem.2014.03.064

Taghvaei, M., Jafari, S.M., Nowrouzieh, S. and Alishah, O. (2015). The influence of cooking process on the microwave-assisted extraction of cottonseed oil. *Journal of Food Science and Technology 52*, 1138-1144. https://doi.org/10.1007/s13197-013-1125-5

Thompson, C.N., Hendon, B.R., Mishra, D., Rieff, J.M., Lowery, C.C., Lambert, K.C., Witt, T.W., Oswalt, S.J., Bechere, E., Wayne-Smith, C., Cantrell, R.G., Kelly, B.R., Imel-Vise, R.K., Chapman, K.D., Dowd, M.K. and Auld, D.L. (2019). Cotton (*Gossypium*

hirsutum L.) mutants with reduced levels of palmitic acid (C16:0) in seed lipids. *Euphytica 215*, 112. https://doi.org/10.1007/s10681-019-2423-4

Tian, X., Ruan J., Huang, J., Fang, X., Mao, Y., Wang, L., Chen, X. and Yang, C. (2016). Gossypol: phytoalexin of cotton. *Science China Life Sciences 59*, 122-129. https://doi.org/10.1007/s11427-016-5003-z

Tian, Y., Xu, Z., Zheng, B. and Martin Lo, Y. (2013). Optimization of ultrasonic-assisted extraction of pomegranate (*Punica granatum* L.) seed oil. *Ultrasonics Sonochemistry 20*, 202-208. https://doi.org/10.1016/j.ultsonch.2012.07.010

USDA (2019). https://downloads.usda.library.cornell.edu/usda-esmis/files/kp78gg36g/h415pj57g/8w32rd83p/cotton.pdf

Wegier, A., Alavez, V. and Piñero, D. (2016). Cotton: Traditional and modern uses. In: *Ethnobotany of Mexico*, (R. Lira, A. Casas, and J. Blancas, eds). Pp. 439-456. Springer, New York.

Wong, S.T., Tan, M.C. and Geow, C.H. (2019). Optimization of ultrasound-assisted ethanol extraction of hazelnut oil. *Journal of Food Processing and Preservation 43*, e14138. https://doi.org/10.1111/jfpp.14138

Yousuf, O., Gaibimei, P. and Singh, A. (2018). Ultrasound assisted extraction of oil from soybean. *International Journal of Current Microbiology and Applied Sciences 7*, 843-852. https://doi.org/10.20546/ijcmas.2018.707.103

vZhang, Q.A., Zhang, Z.Q., Yue, X.F., Fan, X.H., Li, T. and Chen, S.F. (2009). Response surface optimization of ultrasound-assisted oil extraction from autoclaved almond powder. *Food Chemistry 116*, 513-518. https://doi.org/10.1016/j.foodchem.2009.02.071