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RESEARCH ARTICLE

Partial characterization of aerogels made from chayotextle and potato starch

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ABSTRACT

Starch aerogels were obtained from Chayotextle (*Sechium edule*) and potato (*Solanum tuberosum*) tubers. An hydrogel was formed by the gelatinization and gelling of a starch dispersion, subsequently, the exchange of water for acetone yields an acetogel, which is then subjected to a supercritical drying process with CO_2 to obtain an aerogel. In the present work, we used an unconventional starch source, Chayotextle, and compared it with potato starch. A physicochemical and morphological analysis was carried out to evaluate the properties of the obtained aerogels (FTIR, SEM, % Moisture Content, Water Absorption Capacity, Resistant Starch Content, Oil Retention Capacity and Swelling). The results showed that a stirring speed of 800 RPM and 3 days of cooling allowed obtaining aerogels with a homogeneous porous structure. Changes in Moisture Content, solubility, Oil Retention Capacity, Water Retention Capacity and Swelling could be verified in the aerogels obtained. Chayotextle starch aerogels samples have a lower swelling factor and lower moisture absorption compared to potato starch aerogels. This can be used in certain strategic applications. The size of the granules of chayotextle starch allows for a higher content of resistant starch, which shows a greater tendency to retrograde than potato starch. Aerogels can be used for a variety of advanced food applications: from smart ingredients that control nutrient release to active compound delivery systems; from fat substitutes to new biodegradable and smart food packaging materials.

Keywords: Aerogels, Starch, Supercritical drying, Chayotextle, Potato.

INTRODUCTION

Starch is one of the most widely used excipients in the food and pharmaceutical industries where they are used as carrier, thickeners, binders, disintegrants as well as gelling and water retention agents (Soleimanpour et al., 2020). Chemically, starch is a mixture of two very similar polysaccharides, amylose and amylopectin. It polysaccharide, is inert in nature, white in appearance, and relatively tasteless, odorless and low-cost. However, to achieve or eliminate undesirable properties in starch, physical and chemical modifications have been made to alter the granular structure and modify its physicochemical properties (Okolo et al., 2013). Moreover, their natural chemical diversity and characteristic gelling property are particularly attractive for aerogel technology. Aerogels are defined as an open polymeric or colloidal network consisting of loose, packed fibers or particles that are greatly expanded in volume by a gas and exhibit the high porosity required of these materials, very low density and high specific surface area.

The production of biopolymer-based aerogels commonly consist in three steps: hydrogel formation, solvent exchange (usually ethanol), and supercritical drying with carbon dioxide (CO_2) (García-González et al., 2011). The aerogels materials have been prepared from different sources of starches, using several gelation methodologies as thermal, inclusion complexing (Goimil et al., 2017). They can be used in numerous applications, performing different functions, thanks to their peculiar properties such as low thermal conductivity and microporosity. Aerogels have been used as adsorbents, support for catalysts, carrier matrix, thermal and acoustic insulators for buildings (Smirnova and

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Gurikov, 2018). However, the physicochemical properties starch based aerogels are directly correlated with source and concentration of starch, amylose/amylopectin ratio, size of the granules (Druel et al., 2017). Several biopolymers have been reported in form individual or in blends with starch to elaboration of aerogels, (De Marco et al., 2017) finding that the properties of starch-based aerogels are related to the molecular structure of this natural polymer. For the development of these materials it is necessary to have a good intermolecular interaction by hydrogen bonds. Other works reported that amylose/amylopectin ratio in the elaboration of aerogels influence the thermal conductivity transference and that the concentration effect of wheat starch in aerogels, finding that concentration of starch did not affect the internal structure (Ubeyitogullari and Ciftci, 2016). To improve aerogels, new methods have also been developed and new sources of starch could be used (Soorbaghi et al., 2019). Santos-Rosales et al. (2020) elaborated corn starch aerogels as proposals for the use of these materials as scaffolds in tissue engineering due to their high porosity of 85-92%. Other works (Zhu et al., 2018) have focused on obtaining porous microspheres for the encapsulation of active compounds, demonstrating that the process of crosslinking starch with other compounds could improve the integrity and stability of the microspheres.

Chayotextle (*Sechium edule*) is the tuberized root of the chayote plant; it produces tubers that are rich in starch. The properties of chayotextle tuber starch were compared with those of potato starch and reported differences in peak viscosity, gelatinization temperature, and molecular weight (Hernández-Uribe et al., 2011). These differences in molecular structure and physicochemical properties could produce biomaterials with interesting properties as was reported, who finding differences in mechanical, water vapor permeability and thermal properties in biofilms made with chayotextle starch vs potato starch (Aila-Suárez et al., 2013). Other autors reported differences in baked foam materials made chayotextle starch and plantain starch. They reported that molecular differences between these starches influence the biodegradation rate and structural properties. Studies on use of unconventional starches to uses in new applications could be good alternative due to differences in physicochemical and molecular properties (Roman-Brito et al., 2020).

The aim of this work was to investigate the effect of chayotextle starch in the production of aerogels, seeking to provide the starch of this tuber with added value and functionality, since only obtaining foams formulated with this starch has been reported (Vargas-Torres et al., 2017), but not the production of aerogels, which is of great importance. scientific interest, as well as characterize their physicochemical, microstructural properties and resistant starch content. The objective of this research was to compare the properties of aerogels made with potato starch and chayotextle, considering the usefulness of the characteristics of both starches as a possible new material for food and pharmaceutical applications (Lara et al., 2021).

MATERIALS AND METHODS

Chayote (*Sechium edule*) tubers were collected in Tulancingo, Hidalgo, Mexico. Native maize starch was purchased from ALMEX-México (Guadalajara, Jalisco, México); it had an amylose and moisture content of 24% and 11%, respectively.

Hydrogel formation

A dispersion of 8% starch (w/v) in distilled water was formulated in a jacketed cylindrical reactor. The dispersion was heated by means of a water recirculator (PolyScience 812) until it reached a temperature of 90 °C, maintaining constant stirring at 800 rpm for 50 min using a digital mixer (Cole-Parmer 50006-00), see Fig. 1. The gelatinized dispersion was subjected to an autoclave process at a temperature of 121 °C and a pressure of 15 psi (Felisa FE-399) for 20 min. Afterwards, the solution was then transferred to containers that were cooled at 4 °C, allowing the gelled starch to retrograde for a period of three days.

Solvent exchange

The starch hydrogels were cut into monoliths and immersed in acetone, the amount of acetone was six times the volume of the hydrogels and the solvent was changed every 24 h for three days. The material obtained in this process is called acetogel (Druel et al., 2017).

Aerogels formation

The aerogels were made by subjecting the acetogels to supercritical drying, using reagent grade CO_2 at a pressure of 800 psi and at a temperature at 40 °C, so that the CO_2 remained above its critical state. CO_2 outflow was regulated by macro and micrometric valves and maintained constant at 0.5 kgh^{-1}. The drying step was carried out for 3 h. Immediately after, the system was depressurized at the same CO_2 flow rate during 25 min and the dried aerogels were collected and stored in dark Ziploc bag at room temperature (25 °C), for further studies. The images of hydrogels, acetogels and aerogels made based-starch are shown in Fig. 2.

Physicochemical characterization

The physicochemical characterization (swelling factor; solubility; water absorption capacity; and oil retention capacity) was determined following the methodology described with some modifications (Roman-Brito et al., 2020). The dry samples were cut to adjust their weight

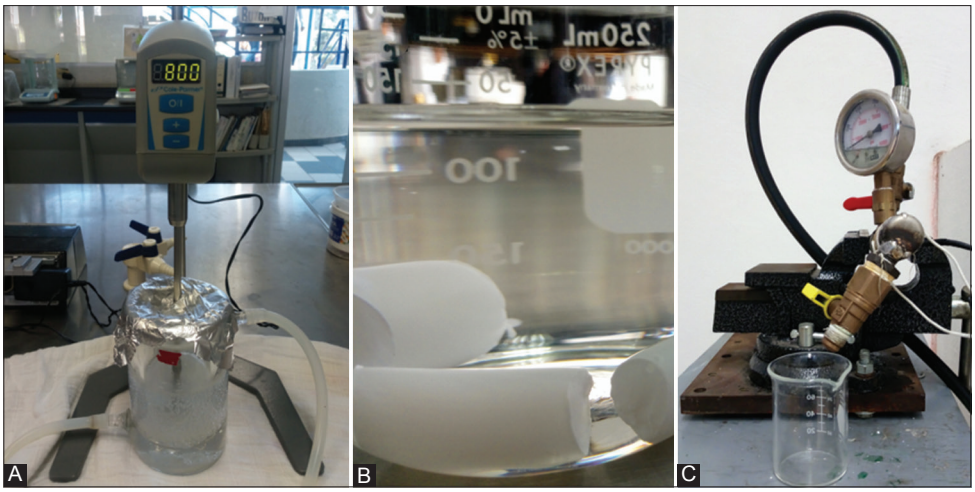

Fig 1. Hydrogel production process (A), solvent exchange (B) and supercritical drying (C) to obtain starch aerogels.

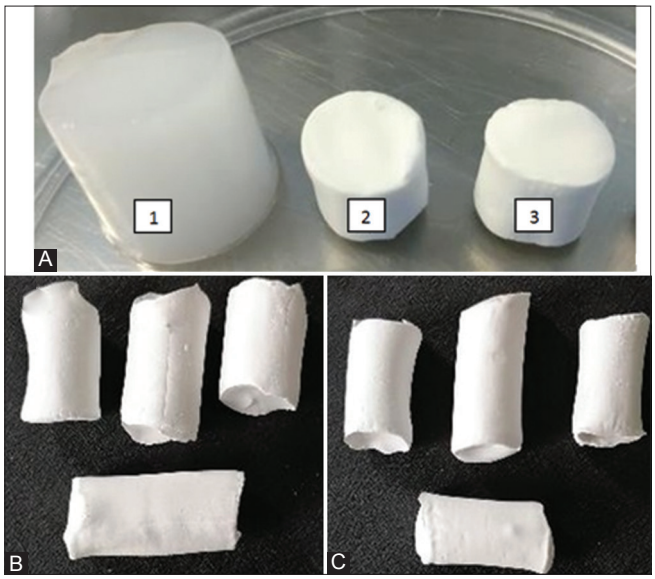

Fig 2. Process for obtaining aerogels is showed in Fig. (A). A1) hydrogel; A2) acetogels; A3) aerogel. The aerogels obtained from the chayotextle and potato starches are shown in Fig.s B and C, respectively.

to 0.3 g and put in centrifuge tubes, then 30 mL water was added. The samples were mixed in a vortex mixer for 30 sec and then heated in a water bath for 30 min under constant stirring using a magnetic stirrer (Thermo SCIENTIFIC). Afterwards, the tubes were centrifuged at 4500 rpm for 15 min (HERMLE Z323K). The weight of the sediments and was recorded. The supernatant in the tubes was dried in an oven at 105 °C for 24 h and its weight was recorded. The same method was used to assess the oil retention capacity of the samples (Fig. 3), using olive oil (Nutrioli) and the weight of the sediments was recording. Moisture content was calculated using the method for dry samples described in the AOAC 925.10 standard. The respective calculations were made according to the following formulas:

Swelling Factor ($g_{ge}1/g_{\text{soluble solids}}$)

$$Swelling\ factor\ (SF) = \frac{Gel\ weight\ (g)}{Sample\ weight\ (g) - Soluble\ solids\ weight\ (g)} \quad (1)$$

Solubilitty ($g_{\text{soluble solids}}/g_{\text{sample}}$)

$$\%Solubiity\ (S) = \frac{Soluble\ solidsweight\ (g)}{Sampleweight\ (g)} x\ 100 \quad (2)$$

Water absorption capacity ($g_{\text{retained water}}/g_{\text{sample}}$)

$$Water\ absorption\ capacity\ (WAP) = \frac{Gelweight\ (g) - Sampleweight\ (g)}{Sampleweight\ (g)} \quad (3)$$

Oil retention capacity ($g_{\text{oil retained}}/g_{\text{sample}}$)

$$Oilretention\ capacity\ (ORC) = \frac{Gelweight\ (g) - Sampleweight\ (g)}{Sampleweight\ (g)} \quad (4)$$

Determination of pore size of aerogels.

Measurement of the most representative pore sizes of a size of 0.5 to 0.9 µm was carried out using the software of the scanning electron microscope, these measurements were extrapolated to measure the visible

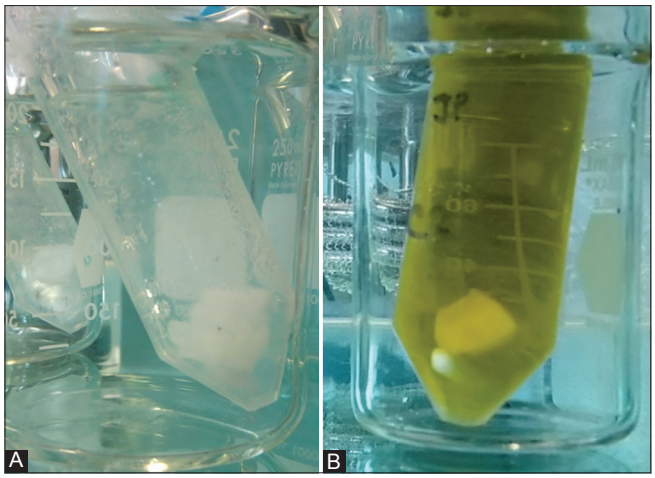

Fig 3. Starch aerogels during the analysis of (A) water absorption and (B) oil retention.

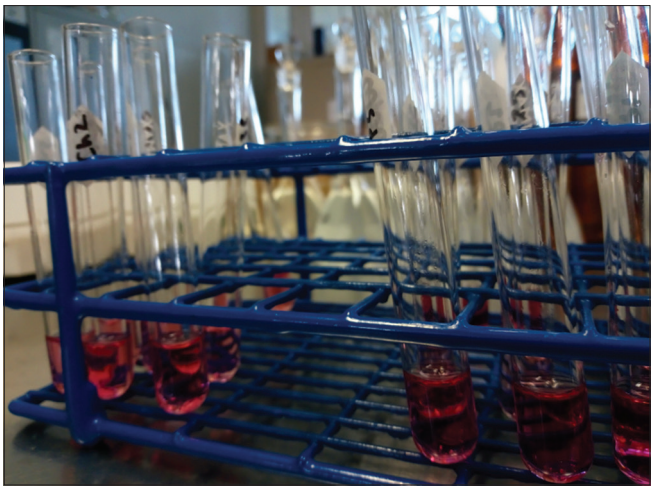

Fig 4. Aerogel samples during the reaction of resistant starch glucose to the GOPOD reagent prior to being analyzed in a UV spectrometer.

pores in the captured micrographs, taking samples from a total of 70 pores measured. From the data obtained, the frequency of the pore sizes was obtained (Ubeyitogullari and Ciftci, 2016).

Resistant starch content in aerogels

Resistant starch (RS) was determined according to the Megazyme procedures (Megazyme International Ireland., 2019). Samples were firstly incubated with 4 ml of pancreatic α-amylase and amyloglucosidase in a shaking water bath for 16 hr. The hydrolytic reaction of starch to glucose was terminated by the addition of pure ethanol, and the RS was recovered as a pellet on centrifugation at 3000 rpm for 10 min. The residue was washed twice by suspension in aqueous ethanol 50% (v/v), followed by centrifugation. RS in the pellet was dissolved in 2 M KOH followed by neutralization and finally the starch was quantitatively hydrolyzed to glucose with AMG. Glucose was measured at 510 nm with glucose oxidase/peroxidase reagent (GOPOD) and calculated as the RS content (Fig. 4).

Fourier-transform infrared spectroscopy (FT-IR)

The powdered aerogel samples and native starches were analyzed using fourier-transform infrared spectrophotometry (Perkin Palmer). The analysis was performed in the spectral range of 4000 to 600 cm^{-1} in order to identify the functional groups present in the samples.

Scanning Electron Microscope (SEM) Analysis

The samples of both aerogels and xerogels were sprayed with gold coating using turbomolecular pumping with a thin film deposition system (DENTON VACUUM Desk V) to avoid problems caused by the presence of static charge in the samples when taking micrographs with the SEM. Once the sample were coated, the aerogels were observed under the scanning electron microscope (JEOL JSM-6010LA).

Statistical analysis

The means (three replicates) and SDs were determined for all determinations. A commercial software program (Sigma Plot ver. 12.5) was used to evaluate by one-way analysis of variance (ANOVA) to determine differences in mean values based on data collected from replications of each measurement. Statistically significant differences ($p < 0.05$) were evaluated using the Tukey multiple comparison procedure.

RESULTS AND DISCUSSION

Physicochemical characterization

Swelling factor (SF), solubility percentage (SP), water absorption capacity (WAC) and oil absorption capacity (OAC) of native chayotextle starch, native potato starch, chayotextle aerogel, potato aerogel and the respective powdered aerogels values are showed in Table 1. The ranges of values observed in the samples for SF study were between 3.41 and 27.97. Chayotextle starch (CS) and chayotextle starch aerogel (CSA) showed low values than observed in potato starch (PS) and potato starch aerogel (PSA). This behavior is related at the chemical composition as reported, (Jayakody et al., 2007) who mention that high lipid contents in starches cause a reduction with the interactions of water molecules, causing a reduction in SF and solubility values (Sol), as observed in Table 1. Other work reported content in fat to chayotextle and potato starch of 0.34 % and 0.24 %, respectively (Hernández-Uribe et al, 2011). These same authors reported that between chayotextle and potato starch there are differences in shape, granule size, crystallinity, and amylose content. Several reports mention that swelling factor in the starches are related with the chemical composition, variation in starch granule

Table 1: Swelling factor (SF), solubility (Sol), water absorption capacity (WAC), oil absorption capacity (OAC and resistant starch (RS) of native chayotextle starch, native potato starch, chayotextle aerogel, potato aerogel and the respective powdered aerogels. All data is expressed as mean ± standard deviation (n= 3). Values with different superscript letters are significantly different (α = 0.05).

8.29 ± 0.73 [c] 2.08 ± 0.15[d]	24.65 ± 0.50[c] 7.63 ± 0.91[d]	Sol	WAC	OAC	Resistant starch (%)
		$g_{soluble\ solids}/g_{sample}$	$g_{retained\ water}/g_{sample}$	$g_{oil\ retained}/g_{sample}$	
Chayotextle starch	3.41 ± 0.12[a]	0.58 ± 0.14[a]	2.39 ± 0.11[a]	2.64 ± 0.07[a]	48.91± 1.95[a]
Chayotextle starch aerogel	6.92 ± 0.24[b]	1.11 ± 0.51[b]	5.84 ± 0.22[b]	2.25 ± 0.10[a,b]	5.17 ± 0.02[b]
Powdered chayotextle starch aerogel	27.97 ± 0.75[c]	8.29 ± 0.73[c]	24.65 ± 0.50[c]	3.92 ± 0.10[c]	
Potato starch	8.81 ± 0.92[d]	2.08 ± 0.15[d]	7.63 ± 0.91[d]	2.44 ± 0.02[a]	62.72 ±2.08[c]
Potato starch aerogel	11.55 ± 1.09[f]	4.44 ± 1.02[e]	10.03 ± 1.04[e]	1.94 ± 0.30[b]	4.46 ± 0.14[d]
Powdered potato starch aerogel	27.24 ± 0.46[c]	19.76 ± 0.37[f]	20.85 ± 0.28[f]	3.38 ± 0.14[c]	

morphology, crystallinity, and amylose content (Naiker et al., 2019; Hu et al., 2019). The SF, solubility, and WAC values increase in chayotextle starch (CSA) and potato starch (PSA) aerogels in comparison with starches sources (Table 1).

These adsorption and solubility properties are important in aerogel samples as reported Franco and De Marco (2020), who reported good adsorption and protection inside the porous structure of aerogels. On the other hand, the powders obtained from the grinding of chayotextle and potato aerogels presented the highest values in this same parameter (SF, Sol and WAC), this may be due to an increase in the chemical bonds of amylose and amylopectin with the water molecules. Other works reported similar behavior in wheat aerogel powders, they mention that increase in values is due to an increase in the amorphous part which is related to the high free energy of amorphous structure (Ubeyitogullari and Ciftci, 2016). However, few studies have been focused on characterizing the powders obtained from aerogels. In this sense, more studies could be focused to understand these behaviors. The water absorption capacity (WAC) showed a slight reduction in the values between starch and aerogels (Table 1), this behavior could be due to the fact that the outer layer of aerogels has low permeability, which causes a decrease in oil-uptake. Highest values were observed in the powders obtained of aerogels, 3.92 to chayotextle and 3.38 to potato. Not observing statistical differences between them (α=0.05). Has been reported that the change in the starch granules from a semi-crystalline state to an amorphous state due a modification would facilitate oil absorption (Chen et al., 2020).

The chayotextle starch aerogels had a resistant starch (RS) content of 5.17%, higher than the potato starch aerogels, with 4.46% RS (Table 1). The resistant starch content of native potato and chayotextle starches was significantly reduced after being subjected to gelatinization, since the RS in the aerogels corresponded to retrograded starch present after the heat treatment used for the production of the aerogels (Tharanathan and Mahadevamma, 2003). The RS value of native potato starch was of 62.72 %, similar value was reported (Goni et al., 1996). In contrast, has been reported that obtained a RS value of 4.4% for potato starch subjected to a heat treatment in an autoclave (121 °C for one hour) (Sievert and Pomeranz 1989). It was also reported a content of RS of 4.19% in chayotextle starch modified by acid hydrolysis, attributing this value to the increase in the crystalline region caused by the acid modification of the starch. Very similar values were obtained in the present work for the potato and chayotextle starch aerogels. It has been report that the content of resistant starch in aerogels made with wheat starch was 13% when using a temperature of 120 °C and 17% when using a temperature of 130 °C, associating this behavior to the fact that the aerogels made at 120 °C had a greater surface area than those made at 130 °C, which could have facilitated the accessibility of the enzymes to the starch matrix (Megazyme International Ireland., 2019). Even though wheat starch has similar amylose content than potato starch, the potato and chayotextle starch aerogels had a lower RS content than those made with wheat starch. This might indicate that the aerogels studied in the present work could have a smaller surface area compared to aerogels made with wheat starch.

Morphology and Pore Size

The aerogels obtained showed a homogeneous microporous structure, as can be seen in Fig. 5, which shows that the aerogels made from chayotextle starch (5A) had a more uniform structure than the potato starch aerogels (5B). In histograms showing the distribution of pore sizes (Fig. 5C), elaborated according with the data reported, (Prin et al., 2012) it can be observed that the size of the pores in the Chayotextle starch aerogels are mostly 0.5 μm in size. Of a total of 70 pores measured in the micrographs, 41% were of this size (0.5 μm), while the percentage of pores between 0.6 and 0.9 μm ranged between 11 and 19%. The potato starch aerogels had pores of similar pore size; 46% of them were 0.5 μm in size, while the percentage of pores between 0.6 and 0.9 μm in size ranged between 9 and 17%. A previous job reported that porous size is related to the solubility and bioavailability of aerogels

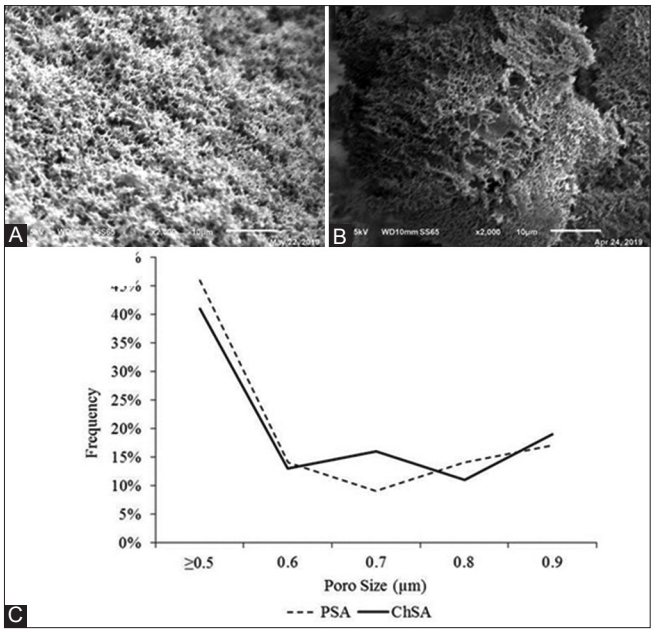

Fig 5. Micrographs of A) a aerogels made from chayotextle starch (ChSA), B) potato starch aerogels (PSA) and C) distribution of pore sizes.

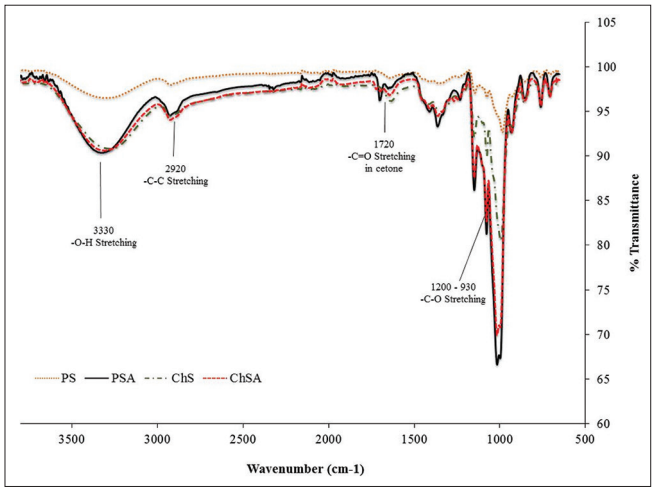

Fig 6. FT-IR chart, where functional groups are visualized and compared aerogel (ChSA) and native chayotextle starch (ChS) and aerogel (PSA) and native potato starch (PS).

(Soleimanpour et al., 2020). Larger porous sizes show good solubility properties. They reported that aerogels with a size of 2-50 nm (mesoporous) are good carriers why have the loading content higher than 10%. As observed in in Fig. 5C, both aerogels in this study could be an excellent material to be used as drug carriers, since shown largest porous size.

FT-IR spectra

The FT-IR spectra of aerogels had more intense peaks than the spectra of native starches Fig. 6). The aerogels showed peaks between 1200-930 cm^{-1}, attributed to the tension and stretching of C-O bonds, as well as to an increase in the transmittance of the aerogels in the signals around the wavelength of 3300 cm^{-1}. These signals are characteristic of starch; the intensity of the peaks at 1047-1022 cm^{-1} is strongly associated with the crystalline and the amorphous regions of starch. It has been reported that, during the retrogradation process, the signals that overlap between 1040 and 1053 cm^{-1} are associated with the development of the helices that form during the first hours of the process and with the aggregation and crystallization of starch molecules. Furthermore, the IR spectrum of starch is sensitive to changes in its structure at the molecular level, such as the formation of starch chains, the formation of helices, crystallinity and the retrogradation process, as well as with water content (Goodfellow and Wilson, 1990; Torres-Becerril et al., 2015). Therefore, an increase in the intensity of the signals of aerogels, compared with the native starches, could be associated with the retrogradation of starch after the it undergoes the changes during associated with the production of aerogels. Was also found signals at 1720 cm^{-1} that are associated with the charge of the carbonyl group in ketones. Similar signals have been reported in taro starch after being modified by acetylation, with a peak at 1750 cm^{-1} (Koga et al., 2003; Cárdenas and Acuña, 2001), which was associated with the vibration of the carbonyl group. In addition to the above, other work reported a decrease in the intensity of the characteristic stretching bands of C=O at 1740 cm^{-1}, and associated it with the interaction between the solvent and the solids. According to Koga et al. (2003), the concentration of a material can be quantified through the absorption force relationship between the number of active infrared bonds in a matrix and the amount of absorption.

In this article, the possibility of using chayotextle starch (unconventional starch source) in the formulation of aerogels is verified, presenting particular characteristics that stand out when compared with potato starch aerogels (Mohammadi and Moghaddas, 2020). The characterization techniques proposed in this work made it possible to demonstrate the efficacy of the parameters selected to obtain the aerogels, in addition to the fact that the production process was carried out in an environmentally friendly manner, since the use of toxic reagents during their preparation. This research opens the door to the study of aerogels made with chayotextle starch in different applications where the microporosity, biodegradability and biocompatibility of this material can be used.

CONCLUSIONS

The technique used for the production of aerogels from potato and chayotextle starch was standardized in the present work. A speed of 800 rpm and 3 days of

refrigeration at 4° C allowed to obtain aerogels with a homogeneous porous structure, with the largest proportion of pores of 0.5 μm in size. The FT-IR spectra of the aerogels showed stronger signals in the 1200-930 cm^{-1} bands compared to the respective native starches. This was due to the formation of helices during the retrogradation process and changes in the crystallinity of the starch. There were also signals in the 1720 cm^{-1} bands due to the presence of acetone residues in the samples. Thus, the FT-IR spectra served as an indication of the efficiency of each drying technique. The starch aerogels showed an increase in their functional characteristics, such as the water absorption capacity, the percentage of solubility, the swelling factor and the oil retention capacity, compared to the native starches of potato and chayotextle. This increase was especially noticeable in powdered aerogels. The values of these parameters in the chayotextle aerogels suggest that the granule size distribution of the native starches influences the functional characteristics of the aerogels produced from them. The chayotextle starch aerogels had a higher content of resistant starch, which might indicate that this starch has a greater tendency to retrogradation than potato starch. The aerogels made in the present work are proof of the potential of both starches to be used in the elaboration of biodegradable materials with multiple functions. The formulation of chayotextle starch aerogels adds value to this type of starch and proves the usefulness of continuing to use and study this starch source. Changes in Moisture Content, solubility, Oil Retention Capacity, Water Retention Capacity and Swelling could be verified in the aerogels obtained. Chayotextle starch aerogels samples have a lower swelling factor and lower moisture absorption compared to potato starch aerogels. This can be used in certain strategic applications. The size of the granules of chayotextle starch allows for a higher content of resistant starch, which shows a greater tendency to retrograde than potato starch. The aerogels obtained in this work presented promising properties since they can be used in the formulation of biodegradable materials with multiple functions within the food and pharmaceutical industry, as well as in packaging systems. The porous structure and solubility position these materials as promising for their use as carriers of active compounds and in tissue engineering. Chayotextle starch is a botanical source of starch that continues to be investigated and gaining importance in the production of biodegradable and biocompatible materials.

Conflict of interest

There are no conflicts of interest between authors or participating institutions.

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Authors contributions

All the authors included in this article contributed equally to the work and their contributions were essential for the completion and preparation of this article. Dr. Rene Salgado Delgado and Dr. Juan Pablo Hernández Uribe are responsible for directing the project and for writing this article. Dr. Heidi María Palma Rodriguez was responsible for monitoring the experimental work. Dr. Alfredo Olarte Paredes and Dr. Areli M. Salgado Delgado served as advisors for the writing of the article and the interpretation of the data, while Ing. Emmanuel Lozano Pineda headed the experimental work of the project in the laboratory and was involved in the writing of this article.

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