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Purification of agave fiber by a hydrothermal method

Purificación de la fibra de agave por un método hidrotérmico

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Abstract

The bagasse of *Agave tequilana Weber* is a lignocellulosic waste generated by the tequila production process. The purification of agave fiber (AF) was carried out by evaluating how the hydrothermal process was affected by temperature and reaction time. Hydrolysis was carried out for 6 h, the combined effects of temperature, pressure and acidification developed within the system caused the release of hemicellulose and lignin from the cellulose microfiber structure coming from agave bagasse. This research work focuses on the study of a hydrothermal process that potentially allows to purify vegetable fibers, providing a good alternative and ecological technique, useful in the manufacture of value-added products, such as polymeric composite materials. Their good mechanical properties and low density of agave fibers (AF) makes them attractive and low-cost materials.

Keywords: agave fiber, microfiber, cellulose, hydrothermal process.

Resumen

El bagazo de *Agave tequilana Weber* es un residuo lignocelulósico generado por el proceso de producción del tequila. La purificación de la fibra de agave (AF) se llevó a cabo evaluando el efecto de la temperatura y el tiempo de reacción del proceso hidrotérmico. La hidrólisis se realizó durante 6 h, la combinación de temperatura, presión y acidificación del sistema provocó la liberación de hemicelulosa y lignina de la estructura microfibrosa de la celulosa que proviene del bagazo de agave. Este trabajo de investigación se centra en el estudio de un proceso hidrotérmico que permite depurar fibras vegetales, aportando una buena alternativa y una técnica ecológica, útil en la fabricación de productos de valor agregado, como los materiales compuestos poliméricos. Las buenas propiedades mecánicas y la baja densidad de la fibra de agave (FA) hacen que sean materiales atractivos y económicos.

Palabras clave: fibra de agave, microfibrilla, celulosa, proceso hidrotérmico.

1 Introduction

Nature provides a large number of useful materials for a wide range of industrial fields, that is why, there is a great interest in investigating such production through biotechnological bioprocesses and research of the production of bioenergy (Montoya-Rosales *et al*., 2019, García-Amador *et al*., 2019), using residues of lignocellulosic materials such as natural fibers, with the aim of minimizing production costs and solve the environmental problems associated with the mismanagement of said waste (Kestur *et al*., 2013), natural fibers such as Sisal (*Agave sisalana*) and henequén (*Agave fourcroydes*) are mainly used as a lightweight structural construction material (Nava-Cruz *et al*., 2015). Agave fibers have certain advantages compared to synthetic fibers, for instance, they have lower density and cost, in addition to being biodegradable and that they can be recycled (Satyanarayana *et al*., 2011, Escoto-García *et al*., 2006).

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One of the most abundant agricultural products in Mexico is the blue agave plant (*Agave tequilana Weber*), and it represents 75% of the agave species that grow in America, also it should be mentioned that 55% of them are endemic (Kestur *et al*., 2013). The average agave production recorded from 2015 to 2018 was 861.65 thousand tons, according to the tequila regulatory council (CRT-Mexico). In recent years, it has been sought to use the natural wastes of companies or industries for the manufacture of novel materials and to reduce the contamination generated nowadays (Rios-González *et al*., 2017), the tequilana agave bagasse is a waste obtained after the plant distillation process takes place in order to produce tequila (Robles *et al*., 2018). Since about a decade ago (Moreno-Vilet *et al*., 2013), a new dynamic field for innovation has focused on the extraction of nanometric size cellulose from lignocellulose (Kalia *et al*., 2011), which is extracted mainly from wood pulp, plants or pure cellulose, the three main chemical fractions that constitute the agave bagasse are cellulose, hemicellulose and lignin (Abdul Khalil *et al*., 2012), they offer a great scientific interest (Singha *et al*., 2014), because they can be useful as a raw material in various synthetic processes (Ruiz *et al*., 2020). Some studies of the types of reactions that can occur during heat treatments, such as depolymerization, demethoxylation, and degradation of sugars have been reported (Garcia-Soto *et al*., 2011). From these constituents, products of greater added value can be obtained with various technological applications (López-López *et al*., 2010). Based on the above, we have investigated the characterization of agave tequilana fibers, obtained after crushing and extracting the cooked pineapple juice from the agave (Kestur *et al*., 2013). Research on plant fibers has increased due to the abundance of these materials. However, the fiber of the agave plant has been poorly studied and of the 256 species registered, only a few have been thoroughly analyzed, among them is the *Agave tequilana Weber* fiber (Hidalgo-Reyes *et al*., 2015). In general, the information reported is about the agave harvest, the use of by-products and parts of the plant, and its characterization is quite limited, due to the abundance and availability of plant fibers in addition to strict environmental regulations (Krishania *et al*., 2018). It has been studied the characterization of plant fiber that is essential to determine the specific properties of bagasse and (Santos *et al*., 2015), in turn, how they are affected by physical and chemical characteristics (Barana *et al*., 2016). Lignin and hemicellulose have been removed very efficiently using acid-alkaline media, but these methods generate toxic environmental residues with high acidity or basicity (Navaee-Ardeh *et al*., 2004). The rupture of the C−O and O−H bonds is responsible for removing lignin during alkaline treatment (Nour *et al*., 2004). These media also work for the removal of impurities such as waxes (Kim *et al*., 2014), pectins and mineral salts increasing afterwards their surface. Acids and alkalis together with pressure can modify the fibers, generating a larger contact surface and reducing their particle size (Naranjo *et al*., 2016).

The most used techniques for the purification or cleaning of different lignocellulosic compounds are based on acid and basic treatments via hydrolysis and the subsequent removal of some components such as: hemicellulose and lignin, which tend to be multi-stage processes with considerable material losses (Abdul Khalil *et al*., 2012). It is of great interest to study and implement a novel purification process for vegetable fibers, specifically the Bagasse *Agave tequilana Weber*, which is a lignocellulosic residue (Yang *et al*., 2012), with great potential for the production of various chemical products with an added value for example bioplastics and biodegradable polymers (Ausias *et al*., 2013). Blue agave (*Agave tequilana Weber*) is used in the tequila industry as a raw material; first agaves are harvested when they reach an average age of 6 to 8 years. After carrying out the leaf cutting (jima), the pineapple is taken to a cooking process and the juice is extracted, which is fermented and distilled for the production of tequila, this process leaves a lignocellulosic residue, the Bagasse *Agave tequilana Weber* (Breton-Deval *et al*., 2018) which maintains a chemical composition of 43% cellulose, 19% hemicellulose and 15% lignin, the annual generation of bagasse is approximately 105 thousand tons. Only a small portion is used to prepare compost, which is why these characteristics make bagasse attractive to be used as a renewable source to produce biofuels and chemicals (Bledzki *et al*., 2015).

An alternative for the purification or cleaning of plant fibers is the hydrothermal process (Rodríguez *et al*., 2009), which is carried out by exerting high pressure inside a Teflon chamber at a programmed reaction time and temperature, this promotes the breaking of internal bonds between hemicellulose-cellulose and lignin-hemicellulose complexes that keep together the microfibrous structure (Torres-Tello *et al*., 2017). The objective of the present investigation was to determine the effect of temperature, the concentration of nitric acid (HNO_3) and the reaction time for the hydrothermal process in order to purify the

agave bagasse and thus obtain clean cellulose fibers, eliminating lignin and hemicellulose and introduce a technique with a greater ecological advantage due to the optimization of the process and the remarkable reduction of environmentally harmful chemicals.

2 Materials and methods

2.1 Raw material

The Agave Fiber (AF) was collected at the distiller (Ex-Hacienda Zacapendo S.P.R. de R.L. Indaparapeo Road - Álvaro Obregón, Km 4.2, Municipality of Indaparapeo, Michoacán de Ocampo, México). Agave bagasse of an age between 6 and 7 years was used, a lignocellulosic residue that can potentially have a high level of aggregated value, so it must be treated and purified to take advantage of its microfibrous structure, for various industrial applications.

2.2 Fiber preparation

The untreated weber agave tequilana bagasse was dried out in the open air (sunlight) for a period of 6 to 7 days. Subsequently, the particle size of the bagasse was reduced manually to approximately 0.5-0.7 cm long, in this way any residue from the crushing process was removed, where the pineapple is squeezed to obtain the useful juice for the preparation of tequila, finally to eliminate moisture as much as possible, it was placed inside the oven at 80 °C, in order to avoid the fiber biodecomposition.

2.3 Chemical composition

The AF were subjected to chemical analysis by determining their content of: ashes by method 31.013 AOAC-1985 (Thamae *et al*., 2007), total reducing sugars (Ting *et al*., 1956), lignin content by the Klasson method (Gassan *et al*., 2001) and cellulose-lignin content. The cellulose content was carried out by difference in cellulose-lignin value determined by the ISO 13906: 2008 method (Sain *et al*., 2006). The determination of the chemical composition was made to the two samples of agave fiber (AF) with hydrothermal treatment and to the (AF) with acid-alkaline treatment with pressure in the same way to the untreated agave fiber AF.

2.4 Hydrothermal treatment

For the optimization of the AF purification process, fiber previously washed with deionized water was used, to remove impurities from the crushing process, a concentration 0.5 M of nitric acid was used for fiber purification, a 45 mL stainless steel autoclave or digester, Parr model 4744, was used, with a maximum working temperature of 250 °C and a maximum pressure of 12410.57 kPa, which promotes the release of organic materials from the AF microfibrous structure such as lignin under environmentally friendly conditions, limiting the use of acidic and basic solutions that are being currently used. Subsequently, it was washed with distilled water to neutralize the solution at pH = 7.0, dried at 120 °C for 2 h to finally characterize the AF with hydrothermal treatment by various techniques.

2.5 Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC)

Thermogravimetric analysis (TGA) was performed using a TGA/DSC analyzer (Perkin Elmer STA 6000, USA) under a constant nitrogen flow (20 mL/min) with a temperature range of 25 to 400 °C. The TGA/DSC was heated from 25 to 400 °C using a heating rate of 10 °C/min; then, the temperature was maintained at 400 °C for 5 min and cooling from 400 to 25 °C at 10 °C/min. The analysis was performed using a 20 mL/min nitrogen flow. Approximately 30 mg of *Agave tequilana Weber* fiber sample was used. Data processing and DTG curves (first derived from the TGA curves, weight loss mg/min) was performed using the Pyris software (Perkin Elmer STA 6000, USA) in similar manner the DSC technique was used to determine the fiber phases transitions.

2.6 Infrared spectroscopy (FT-IR)

The analysis for the Untreated Agave Fiber (UAF) and AF were previously dried at 120 °C for 2 h, to eliminate material moisture; they were analyzed using an IR spectrometer with Fourier Shimadzu IRTrace-100, Japan, and transformed using Attenuated Total Reflectance (ATR). This is done at room temperature 25 °C, with 12 scans, a resolution of 4 cm^{-1} and a wavenumber range of 4000-500 cm^{-1}.

2.7 X-ray diffraction (XRD)

The UAF and AF samples with treatments were analyzed using a Rigaku SmartLab X-ray diffractometer, with graphite monochromator with Cu-Kα1 radiation (λ = 1.54 Å), a Ni filter and a scintillation counter as a 35 kV detector and 30 mA in rotation from 10 to 85 ° at a scale of 2θ according to the technique described by Singha and Rana (2010). The samples were spread and homogenized before being subjected to X-ray exposure. The crystallinity index (C.I.) was determined using diffraction counts at an angle 2θ close to 15 and 22 °, which was calculated based on the following formula:

$$\text{Cristallinity index} = \frac{I_c - I_a}{I_c} \quad (1)$$

where: I_c and I_a are the crystalline and amorphous intensities at a 2θ scale close to 15 and 22 °.

2.8 Morphological characterization by scanning electron microscopy (SEM)

The fiber morphology of the UAF and AF samples with treatments were analyzed using an electronic scanning electron microscope (XL 30 Philips, England) with tungsten filament, at a pressure of 0.94 torr, at room temperature and a resolution 3.5 nm. The material chemical composition was corroborated by EDS using a sample that is copper metallized for 15 seconds. The chemical composition of the UAF and AF with treatments were determined through a dispersive energy X-ray spectrum detector (Edax, USA) coupled to the environmental electronic microscope, using the "Microanalysis System Software" version 3.31XL.

3 Results and discussion

3.1 AF composition

A series of processes were previously carried out based on conventional treatments and that have been reported in the last decade for cleaning or purification of plant fibers, mainly sugar cane fiber and fibers of various agave species, with the aim of having a comparative target of vegetable fiber used in our research.

The fiber analysis of the bagasse of UAF and AF with treatments, showed a very different chemical composition under the applied treatments. The UAF have a chemical composition of 18.56 ± 1.2 w/w of hemicellulose, 49.26 ± 5.31% w/w of cellulose, 28.42 ± 1.7% w/w of lignin and 3.76 ± 0.47% of ashes, while the Agave Fiber with hydrothermal process (FACT) have 20.2 ± 1.4%, w/w of hemicellulose, 48.94 ± 5.27% w/w of cellulose, 25.02 ± 1.12%, w/w of lignin and 5.84 ± 0.7%, w/w of ashes, while for AF-alkali with pressure, the variations are more significant in cellulose loss (table 1). This composition is similar to that reported (Saucedo-Luna *et al*., 2011), under conventional alkaline acid methods. The kinetics of thermal hydrolysis of fructans from other Agave species has been determined during the cooking stage of production of mezcal in a pilot autoclave at different temperatures and cooking times, ranging from 96 to 116 °C and from 20 to 80 h.

Table 1. Chemical composition of AF, acid-alkali AF with pressure and AF hydrothermal process.

Sample	Lignin (%)	Cellulose (%)	Hemicellulose (%)	Ashes (%)
Agave tequilana *Weber* UAF untreated agave fiber	28.42±1.7	49.26±5.31	18.56±1.2	3.76±0.47
Agave tequilana *Weber* AF acid-alkali whit pressure	57.4±5.97	32.41±1.7	5.84±0.7	4.35±0.61
Agave tequilana *Weber* AF hydrothermal process	25.02±1.12	48.94±5.27	20.2±1.4	5.84±0.7

A simple kinetic model of the depolymerization of fructans to monomers and other reducing sugars and of the degradation of reducing sugars to furans [mainly 5-(hydroxymethyl) furfural, HMF]. It has been indicated that the thermal hydrolysis of some varieties of agave, from the ethanol yield point of view, corresponded to a hydrolysis of fructans, producing syrups with furan and concentrations of reducing sugars respectively (Garcia-Soto *et al*., 2011).

3.2 AF with hydrothermal treatment

For the treatment of AF, a high pressure stainless steel autoclave was used, a series of tests (Table 2) using 3.5 g of AF previously washed with the purpose of removing the remaining impurities from the bagasse crushing process; a 40 mL solution was added to 0.5 M HNO_3, performing a series of tests with temperatures varying from 120 to 180 °C and various reaction times from 2 to 8 h, within the system a pressure of up to 12410.57 kPa is reached. The temperature and reaction time for the hydrothermal treatment were standardized, it was found that the optimal conditions for a total cleaning of the AF without degrading its fibrous structure, are those at: 140 °C (7997.92 kPa) and reaction time of 4 h, obtaining a total weight loss of 45 - 52%, attributed to components such as: hemicellulose, lignin and cellulosic waste (Langhorst *et al*., 2019). It can be deduced based on what is reported in the literature, that thermal hydrolysis was achieved at different temperatures and hydrothermal treatment times, which range between 120 to 180 °C and reaction times around 2 to 8 hours, this produces depolymerization from fructans to monomers and other reducing sugars and from the degradation of reducing sugars to furans [mainly 5- (hydroxymethyl) furfural, HMF]. The thermal hydrolysis of the agave fiber favors the subsequent fermentation of alcoholic compounds such as ethanol as well as the breaking of internal bonds of cellulose and lignin components, which is indicative of the whiteness presented during the hydrothermal process of the agave fiber (Garcia-Soto *et al*., 2011).

3.3 TGA thermogravimetric analysis

The thermogravimetric analysis was carried out to characterize the obtained fibers as a function of temperature, as well as to evaluate the effect of the treatments applied. In figure 1, thermograms referring to UAF, hydrothermal process AF, acid-alkali AF are shown. The thermogram curve of the UAF presents a thermal decomposition in the range of 104 to 145 °C due to a moisture loss in the sample with a decrease in its mass between 6 - 7%. The fiber stabilization temperature is found at 150 °C having a weight loss of 10 - 12%, which is attributed to fructans leftovers that are stable at temperatures below 200 °C depending on their branching chains.

Table 2. Standardization of the hydrothermal treatment of the BATW, varying temperature and reaction time of the process.

Reaction time (h)	**Temperature (°C)**	**Weight (g)**	**Volume of H_2O (mL)**	**Process results**
2	120	3.5	40	No reaction
4	120	3.5	40	No reaction
8	120	3.5	40	Minimum whiteness
2	140	3.5	40	No reaction
* 4	140	3.5	40	High fiber whiteness
8	140	3.5	40	Fiber degradation
2	160	3.5	40	No reaction
6	160	3.5	40	Fiber degradation
8	160	3.5	40	Fiber degradation
2	180	3.5	40	Minimum whiteness
4	180	3.5	40	Partial fiber degradation
8	180	3.5	40	Fiber degradation

*Optimal conditions for obtaining clean fiber.

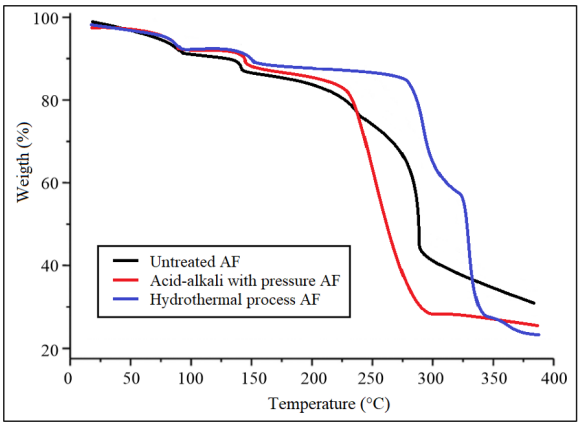

Fig. 1. TGA of *Agave tequilana Weber* fibers, with their untreated AF treatments, acid-alkali with pressure AF, hydrothermal process AF.

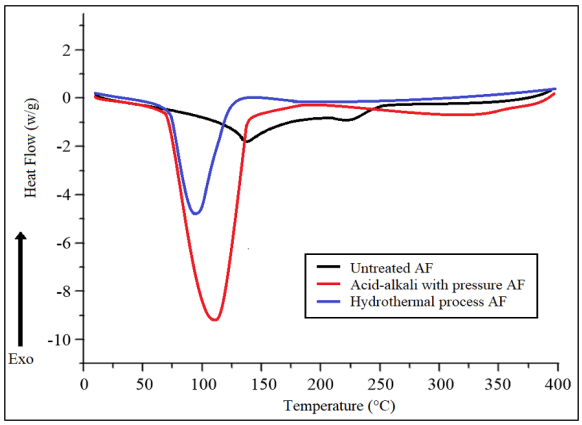

Fig. 2. DSC analysis of untreated AF, acid-alkali with pressure AF with pressure and hydrothermal process AF.

Hemicellulose begins its degradation from 160 °C to 320 °C, in the range 270 - 290 °C it begins to have a considerable weight loss according to the thermogram, this indicates that the fiber has a thermal stability up to 305 °C with a mass loss of 23% (Espinosa-Andrews *et al.*, 2012), after 350 °C, total cellulose degradation is carried out, which means that the microfibrous structure is completely decomposed. The TGA obtained from the fiber before and after the treatment (Figure 1) were compared, corroborating the removal of the lignocellulosic components as well as the physisorbed water by the material fibrous structure, after performing the drying treatments a decrease of total moisture 2 - 2.5% can be observed in the sample. The AF acid-alkali has a considerable weight loss, which is attributed to the lower amount of cellulose which indicates that the obtained material by this method has a more fragile structure possibly due to the degradation of the material microfibrous structure.

3.4 DSC analysis

The heat difference between the agave tequilana fiber before and after the hydrothermal treatment for cleaning was analyzed (Figure 2). The untreated AF and the references acid-alkali with pressure AF and hydrothermal process AF are maintained at the same temperature during the experiment, in order to obtain the minimum changes of the material and identify any of the following physical properties of the material: enthalpy, fusion energy, specific heat, crystallinity, reaction enthalpy, thermal stability, oxidation stability, aging, degree of purity, phase transformation, eutectic compounds, polymorphs and product identification.

One of the main differences between the treated and untreated fiber is that in the first one the enthalpy is lower if compared to the native fiber, probably due to the elimination of larger sugar chains via hydrolysis that takes place during the purification process which can be clearly observed in acid-alkali AF.

Based on the spectrogram shown in figure 2, it is determined that the untreated fiber has an endotherm at 110.73 °C and one more at 213.82 °C, a small plateau at 139.32 °C is also observed, corresponding to the thermal degradation products such as: moisture, reducing sugars, cellulose hemicellulose and lignin. On the other hand the fiber that was treated hydrothermally presents similar peaks but of much greater intensity, an endotherm is observed at 96.9 °C and a maximum plateau at 117 °C due to reactions of substances derived from the degradation of the compounds eliminated during the hydrothermal process, below 130 °C, which is a loss of moisture specifically attributed to fructans, fructose polymers derived from the sucrose molecule, (disaccharide of fructose and glucose). A calorimetric constant of 0.06579 was determined based on the area under the observed curve. Finally, for the acid-alkali with pressure AF, the peak present at 118 - 120 °C has a much greater intensity, which corroborates the considerable loss of cellulose determined in the chemical composition of said material.

3.5 Fourier transform infrared spectroscopy (FTIR)

In the FTIR spectra, the chemical analysis is shown by identifying the main functional groups present in each sample with and without treatment.

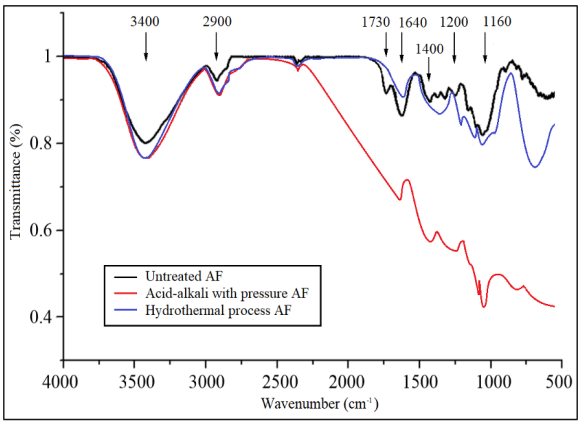

Fig. 3. FTIR spectra of the untreated AF, acid-alkali with pressure AF and hydrothermal process AF

Figure 3 presents an absorption band between 1160 - 1170 cm^{-1}, which is attributed to the asymmetric stretching C−O−C, in the case of hydrothermal process AF a lower signal with respect to the untreated AF is observed (Åkerholm *et al.*, 2001), the band between 3400-3460 cm^{-1} is attributed to the hydrogen bond elongation in −OH, which is observed with less intensity in the AF, this is attributed to the subsequent drying process of the material, which is similar for acid-alkali with pressure AF, the band of 1640 cm^{-1} is related to the amount of water absorbed, originated by the elongation of hydrogen bonds and the flexion of −OH altogether with the cellulose structure, which is consistent with that reported by Mondragon *et al.*, (2014) this reflects that the acid-alkali AF tends to physisorb a larger amount of moisture. The decrease in the intensity of the band at 2900 cm^{-1} indicates that, there is a smaller proportion of cellulose due to a small amount of cellulose loss in the process, in the same way it is corroborated with the intensities for the vibration bands −H and C−O present in 1060 - 900 cm^{-1} which decreased mainly in acid-alkali with pressurized AF, in this way corroborating that the hydrothermal process AF preserves more efficiently the cellulosic material structure with respect to acid-alkali with pressure AF.

The intensity band observed at approximately 3420 cm^{-1} corresponds to the C−H and O−H groups described as ways of lengthening the hydrogen bonds and flexing the hydroxyl (−OH) groups present in the cellulosic material. At 1735 cm^{-1}, an elongation vibration of the C=O groups of the residual of acetyl and uronic pectin esters, hemicellulose or ester bonds of the carboxyl groups in ferulic and *p*-coumaric lignin acids and hemicellulose (Panthapulakkal *et al.*, 2006). The band of 1730 cm^{-1} corresponds to the vibration of the C=O groups attributed to the breakdown of the acetyl bond between lignin and hemicellulose, this is consistent with the high pressures used during the purification processes especially for acid AF alkali, the latter is not very favorable. Finally, the 1200-900 cm^{-1} bands range, refers to a decrease of C−OH and C−O groups from the cellulose, in the acid-alkali AF a much greater loss is observed due to the usefulness of the solvents and pressure exerted during the purification process, allowing cellulose chain cleavages to take place, on the other hand in the AF hydrothermal process the mentioned cellulose loss is not observed as reported in table 1, which indicates that the microfibrous structure of AF does not decompose but only releases components such as lignin and hemicellulose from its fibrous structure.

3.6 X-ray diffraction

X-ray diffractograms in Figure 4 show two phases. The AF were found to include a mixture of β cellulose and whewellite, a crystalline form of oxalate monohydrate of calcium ($CaC_2O_4 \bullet H_2O$), from the acid-alkali process. Whewellite is present mainly as a fine powder (confirmed tangentially by XRD of a screened sample), while the fibers were quite thick (2-4 millimeters in length). As such, a quantification of phase abundance would be misleading due to the difficulty of preparing the samples consistently. The heat treatments did not introduce or eliminate any phase. The diagram indicates that the whewellite phase is clearly affected by the heat treatment of acid-alkali AF with pressure, in the form of extension or widening of the peaks (Figure 4).

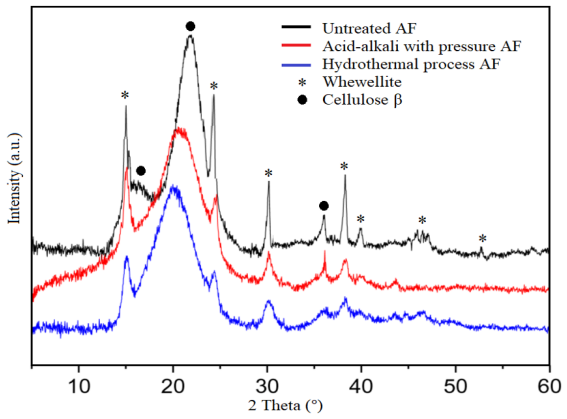

Fig. 4. Difractograms of the untreated AF, acid-alkali with pressure AF and hydrothermal process AF.

Specifically, the peaks are extended with the heat treatment of the hydrothermal process AF and the enlargement increases depending on the temperature and pressure of the treatment. The X-ray diffraction patterns for the UAF, hydrothermal process AF and acid-alkali AF with pressure are shown in Figure 4, respectively. Both residues show a broad peak at low angles. The main diffraction peaks (2θ=14.99 °, 16.49 ° and 22.78 °) correspond to the cellulose structure based on the crystallographic record (JCPDS 50-2241). Figure 4 also shows other diffraction peaks that correspond to the calcite structure $CaCO_3$ (JCPDS 05-0586).

3.7 Scanning Electron Microscopy (SEM)

Based on the micrographs shown, the untreated AF has a very characteristic morphology with longitudinal uniform roughness and in a compact manner with an uniform porosity and an internal pore diameter ranging between 2-6 μm, the observed external thickness was between 20 - 30 μm figures 5-a and 5-b, respectively. It can be seen that the untreated AF maintains a compact structure with relative porosity distributed in the fibrous structure unlike the hydrothermal process AF (Figs. 5-c and 5-d), where a more distributed porosity is observed due to the absence of compounds such as lignin and hemicellulose, so it has a more variety in its porosity. The fiber (Figs. 5-e and 5-f) shows a rough surface in the form of folds suitable to support or anchor various materials that could be useful in filtration processes or as catalytic supports, this based on the microfiber structure shown, it is important to note that the fiber maintains its longitudinal structure and has not degraded. On the other hand, it can be verified that the process of fiber purification by the hydrothermal method shows more favorable results for cleaning since it maintains the microfibrous material structure, obtaining this way a fiber useful in the synthesis of novel materials that require porous surfaces; which can be doped for use in different purification processes or other applications. The chemical composition of AF is corroborated by the EDS technique, determining 84% carbon which is attributed to the chemical composition of cellulose, which was also determined by XRD and TGA, the remaining 16% is attributed to the presence of functional groups such as (−OH, −COOH, −COH or epoxy groups) identified by FTIR.

The SEM images allow us to observe the agave fiber as a structure assembled in several layers, which is similar to other fibers reported by other authors (Agave Americana and *Agave tequilana Weber*, blue variety) (Bessadok *et al*., 2009). Under the lowest hydrothermal treatment conditions, the fiber structure showed no change and retained its structural bundle arrangement with no apparent damage (Figs. 5b-5c). This is evidence of an ineffective fibers delignification. Figures 5d - 5e show SEM images for the hydrothermal process carried out at 140 °C, on them the highest whiteness and no decomposition of the fibrous material are observed, those are corroborated also by XRD; the collected diffractogram indicates that the material maintains its base structure without degrading the fiber cellulosic portion. In figure 5e it is confirmed that fiber structural changes take place: damage to cell structure, cell wall collapse, presence of fractures, clusters of broken fiber and as a result a more porous structure, as well as the presence of structural fiber residues (Figs. 5e-5f); which is similar to that reported by some research groups (Kestur *et al*., 2013).

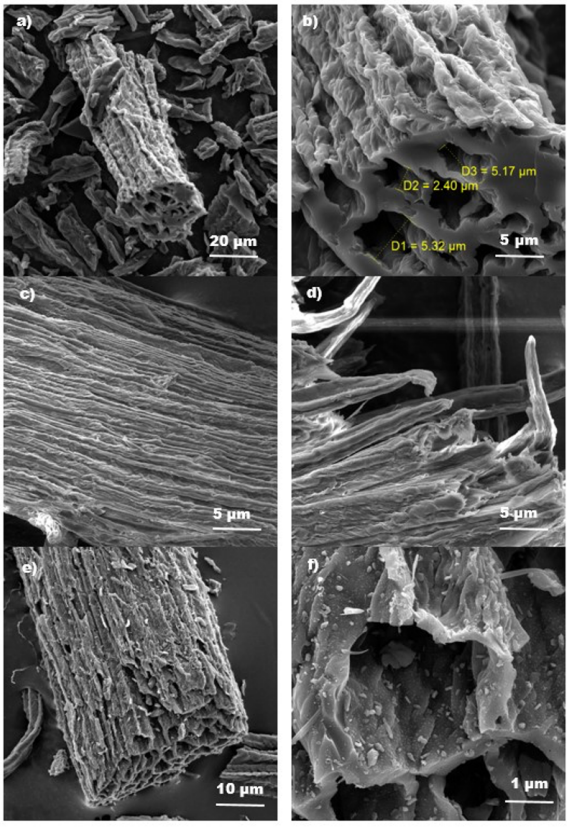

Fig. 5. Micrographs of the untreated AF (a-b), acid-alkali with pressure AF and hydrothermal process AF (c-d).

Conclusions

The development of optimal conditions for the purification process of *Agave tequilana Weber* fiber were achieved by implementing a method aiming to minimize the use of acid or basic processes with lignocellulosic residues highly contaminated. The hydrotreatment process reported provides some more environmentally friendly conditions and presents an excellent option for cleaning of the agave fiber microfibrous structure, which was corroborated by the SEM micrographs and other characterization techniques performed. This novel purification technique for Weber tequilana agave fiber could also be applied to various types of vegetable fibers to remove lignin and hemicellulose from the fibrous structure of bagasse, moreover, introducing a technique with a greater ecological advantage due to the optimization of the process and the remarkable reduction of environmentally harmful chemicals. With the knowledge of various properties, particularly thermal properties, it should be possible to use these AFs to be incorporated into polymers, including biodegradable ones. Therefore, in view of the abundant availability of AF waste in Mexico, a potential composite material could be produced, which could be compostable and biodegradable as well as added value for the waste.

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