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Application of Fourier transform infrared spectroscopy (FTIR) in combination with attenuated total reflection (ATR) for rapid analysis of the tequila production process

Aplicación de la espectroscopía infrarroja por transformada de Fourier (FTIR) en combinación con la reflectancia total atenuada (ATR) para un análisis rápido del proceso de producción de tequila

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Abstract
The manufacturing process of tequila made of *Agave tequilana* Weber var. azul consists of several important process stages such as milling, cooking, fermentation, distillation, and aging. The purpose of this contribution was to characterize five of the most important stages of the tequila production process in a pilot plant by using FTIR-ATR spectroscopy. The results showed that FTIR-ATR spectra of raw juices in the range 4000 and 700 cm^{-1}, were mostly composed of absorption peaks associated with water and fructose. The spectra of cooked juices showed the thermal hydrolysis of the fructans to fructose in the interval between 1200 and 800 cm^{-1}. The FTIR-ATR spectra of the samples fermented showed a gradual sequence transforming fructose peaks to ethanol peaks during the interval time from 0 to 48 h at 35°C. The spectra obtained from the distillation showed that the intensity of the peaks was a function of the concentration of ethanol in the product distillates. The evolution of the aging of the "*tequila blanco*" in an oak barrel was monitored for 60 days. The results showed that the region between 1300 and 900 cm^{-1} of the FTIR-ATR spectra was associated with the aging process in the oak barrel.
Keywords: tequila, FTIR-ATR spectroscopy, agave juice, cooking and fermentation process, stage of aging.

Resumen
El proceso de elaboración del "Tequila" a base de *Agave tequilana* Weber var. azul consta de varias etapas importantes de proceso tales como la molienda, cocimiento, destilación, y añejamiento. El propósito de este trabajo fue caracterizar cinco de las etapas importantes del proceso de producción del tequila en una planta piloto mediante espectroscopía FTIR-ATR. Los resultados mostraron que los espectros FTIR-ATR de jugos crudos se presentaron en el intervalo entre 4000 y 700 cm^{-1} donde están solamente los picos de absorción asociados con sus compuestos principales agua y los fructanos de agave. Los espectros de jugos cocidos mostraron la hidrólisis térmica de los fructanos a fructosa en el intervalo entre 1200 y 800 cm^{-1}. Los espectros FTIR-ATR de las muestras fermentadas mostraron una secuencia gradual de transformación de los picos de fructosa en picos de etanol durante un intervalo de tiempo de 0 a 48 h a 35°C. Los espectros obtenidos de la destilación mostraron que la intensidad de los picos era función de la concentración de etanol. Se dio seguimiento de la madurez del "*tequila blanco*" en barrica de roble durante 60 días, finalmente, los resultados mostraron que la región entre 1300 y 900 cm^{-1} de los espectros FTIR-ATR se asoció con el proceso de maduración en la barrica de roble.
Palabras clave: tequila, espectroscopía FTIR-ATR, jugo de agave, proceso de cocimiento y fermentación, etapa de maduración.

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1 Introduction

Tequila is a Mexican alcoholic beverage widely known around the world, which is produced from the heads of *Agave tequilana* Weber var. azul. Tequila production begins when *A. tequilana* plants are harvested at the optimum maturity age, generally between 6 to 8 years. The juices of the heads are rich in agave fructans, which are polymer chains composed of units of fructose linked to a molecule of sucrose (López *et al*., 2003, Rodríguez-Garay, 2015). At industrial level, the agave heads are cooked to hydrolyze the juices rich in agave fructans to fructose (Waleckx *et al*., 2008). These fructose juices are first fermented, followed by a double-distillation to obtain tequila, called "*tequila blanco*" (Prado-Ramírez *et al*., 2005). The "*tequila blanco*" (white tequila) is a mixture of ethanol and water with traces of higher alcohols, aldehydes, fatty acids, esters, sulfur compounds, etc. (Prado-Jaramillo *et al*., 2015, Lopez-Ramirez *et al*., 2013, Martin del Campo *et al*., 2011), giving the characteristic aroma and flavor to the beverage. Tequila can also be later aged in oak barrels to produce "*tequila reposado*" (tequila rested up to three months in barrels) or "*tequila añejo*" (aged tequila, with 1 to 5 years in barrels).

The quality of tequila in its different step productions has been studied using Gas Chromatography (López-Ramírez *et al*., 2013, Arellano *et al*., 2008, Arrizon *et al*., 2006, Vallejo-Cordova *et al*., 2004) or High Performance Liquid Chromatography (HPLC) (Muñoz-Muñoz *et al*. 2008, Muñoz-Rodríguez *et al*., 2005), Raman spectroscopy (Frausto-Reyes *et al*., 2005), UV-Vis spectroscopy (Contreras *et al*., 2010, Barbosa-García *et al*., 2007), and Fourier Transform Infrared spectroscopy (FTIR), in the infrared medium region (4000-650 cm^{-1}) (Smyth and Cozzolino, 2013, Lachenmeier *et al*., 2005). The FTIR spectroscopy in combination with the ATR interaction accessory, has been widely used to characterize various types of biopolymers (Arrieta-Almario, *et al*., 2019) as well as alcoholic and non-alcoholic beverages (Gómez-Montaño *et al*., 2021, Belchior *et al*., 2019, Lohumi *et al*., 2015, Silva *et al*., 2014, Leopold *et al*., 2011, Karoui *et al*., 2010, Llario *et al*., 2006, Paradkar *et al*., 2002). FTIR spectroscopy features the following advantages: fast response to obtaining spectra, for the analysis it is only needed a small amount of the sample, it is low-cost equipment, it has high repeatability, and often it does not require chemical agents for the preparation of the tested sample. In the ATR device, the sample is placed on the surface of a crystal with a high refractive index where the infrared radiation interacts in an attenuated form inside the sample (Lee *et al*., 2017, Rodriguez-Saona and Allendorf, 2011, Hind *et al*., 2001). The spectrum obtained from the FTIR-ATR measured in the range 4000 to 650 cm^{-1} (mid-infrared), provides information related to the vibrational modes of the bonds of a sample (for example, stretching or bending) (Baishya *et al*., 2021, Stuart, 2004, Coates, 2000). In this contribution, it is reported the rapid and reliable monitoring of the tequila process using FTIR-ATR spectroscopy. The different stages contemplated ranged from the characterization of the raw juices of the agave head, followed by the cooking, fermentation, distillation, and aging stages in a pilot plant process. The FTIR-ATR spectra will allow determining the state of the production process in its different stages. If the production samples are measured in real-time important decisions could be taken opportunely during the production process.

2 Materials and methodology

2.1 Material

The plant heads of *A. tequilana* Weber var. azul at the optimum age (6 years) were purchased from producers in the town of Tequila, Jalisco, Mexico (Latitude: 20° 52’ 34.79" N, Longitude: 103° 50’ 7.19" W).

2.2 Samples of the tequila process

The raw juice was obtained by grinding the *A. tequilana* Weber var. azul heads. The raw juice was immediately characterized by FTIR-ATR spectroscopy. In the cooking stage, the agave heads were split in halves and subjected to a cooking process for 6, 12, 24, 36, and 48 h at 95 °C. Juice samples were collected by grinding the cooked heads. The juice extracted was stored at 4 °C until analysis. For the fermentation stage, the yeast *Saccharomyces cerevisiae* was grown in the agave cooked juice at a temperature of 35°C for 48 h. The fermentation process was sampled every 8 h.

The distillation process was carried out in two stages: in the first one an ordinary product with content between 20 and 30% of alcohol volume was obtained. In the second stage, a distillate between 40 and 60%

volume of alcohol was achieved. Heads and tails in both cases were discarded. The samples selected in duplicate were stored under refrigeration at 4 °C until the characterization by FTIR-ATR spectroscopy.

2.3 FTIR-ATR spectroscopy

The FTIR-ATR spectra from all samples were obtained using an infrared spectrometer Cary 630 (Agilent, USA) equipped with an ATR accessory of a single internal reflection. Samples were placed on diamond/ZnSe crystal plate of 1.8 mm in diameter and scanned from 4000 to 650 cm^{-1} for 20 scans with a resolution of 4 cm^{-1}. Previously for each spectrum, the background of the surrounding air was collected. The spectrum of water was subtracted from each spectrum obtained from the cooking and fermentation stages. All the spectra were plotted in function of the wavenumber (cm^{-1}) versus the absorbance.

2.4 Aging process

A white oak barrel was purchased from artisan manufacturers in the tequila region of Amatitán, Jalisco, Mexico. The capacity of the barrel was 5 liters and had internal toasting carried out by the manufacturer. The "*tequila blanco*" had an alcoholic strength of 35% alcohol vol. The "*tequila blanco*" was placed in the barrel up to 90% of its capacity and stored at 20 °C. During the experiment, a sample of mature tequila was taken from the barrel every 10 days for 60 days. FTIR-ATR measurements were taken for every sample to obtain their spectrum. In this step, each sample obtained was placed on the glass of the ATR accessory (Approximately 19.5 mg of the sample). The tequila sample was allowed to evaporate at room temperature to eliminate the ethanol and water. The acquisition of the corresponding FTIR-ATR spectrum was carried out by observing the disappearance of the peaks associated with ethanol and water. Before obtaining the infrared spectra, the background of the surrounding air was obtained, which was subtracted automatically during the measurement of each sample.

3 Results and analysis

3.1 FTIR-ATR characterization of the raw agave juices

The FTIR-ATR spectrum of the raw agave juice showed peaks associated with its major components: water and agave fructans as shown in Figure 1. In the spectral range, located between 1200 and 800 cm^{-1}, the peaks that appear come from bonds of the agave fructans molecules (Vázquez-Vuelvas *et al*., 2020). Around 1024 cm^{-1} appeared an intense peak associated with the C-O stretch of the C-OH group and the C-C stretch of the fructans. The peak at 1128 cm^{-1} was associated to the stretching of the C-O bond of the C-O-C linkage. The peak located at 928 cm^{-1} was due to the bending of the C-H bond of the agave fructans. The peak located at 2940 cm^{-1} was assigned to the stretching of the C-H bond from CH_2 and the peak at 2880 cm^{-1} was related to the C-H bond from CH_3. Two peaks associated with the water molecule located around 3245 and 1636 cm^{-1} were observed in Figure 1, mainly caused by vibrations of stretching and bending at the bonds O-H and H-O-H, respectively (Stuart, 2004). In infrared spectroscopy, water is the majoritarian component of the agave juice samples; therefore, the infrared modes of water are very intense and may overlap with the signals of interest. Nevertheless, in this study, the water peaks do not cover the characteristics peaks of the agave fructans in the region 1200 to 800 cm^{-1} (Table 1).

Table 1. Bond and their mode of vibration obtained from the FTIR spectra of raw agave juice.

Wavenumber (cm^{-1})	**Bond**	**Mode of vibration**
3245	O-H (water)	stretching
2940	C-H (CH_2) (agave fructans)	stretching
2880	C-H (CH_3) (agave fructans)	stretching
1636	H-O-H (water)	bending
1500-1200	skeletal vibration (agave fructans)	bending/stretching
1200-900	C-O, C-C (agave fructans)	bending/stretching

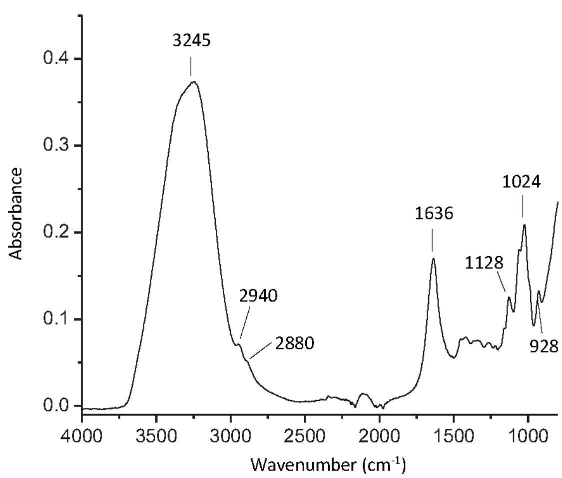

Figure 1. Representative FTIR-ATR spectrum of raw agave juice in the 4000-750 cm^{-1} spectral region.

Hence, the FTIR-ATR technique can be associated with the maturing process of the agave given that the intensity of the peaks located in the region between 1200 and 800 cm^{-1}, is the consequence of the formation of fructans in the agave heads (Mellado-Mojica and Lopez, 2012).

3.2 *FTIR-ATR spectroscopy characterization of the cooked agave juices*

During the cooking of the agave heads, the fructans are mainly converted to fructose, with a lower percentage of glucose (<10%). Traditionally, the cooking of the agave is carried out in masonry ovens at a temperature of 95 °C for a period time between 28 and 48 h. Nowadays, to reduce the duration of cooking, agave heads are usually cooked in autoclaves applying steam under pressure, reaching temperatures about 120 °C, during a time interval of 12 to 24 h. Cooked juices have been characterized by UV spectrometry (at 490 nm), °Brix, HPLC quantification of fructose and glucose, and changes in the pH of the juices depending on the temperature and the cooking time (Waleckx *et al*., 2008).

In infrared spectroscopy, every carbohydrate has characteristic peaks associated with stretching vibrations of C-O, C-C, or C-O-C in the range from 1400 to 800 cm^{-1}, where its position and intensity change depending on the type of carbohydrate analyzed (Cassani *et al*., 2018). Notably, a maximum peak of fructose dissolved in water approximately at 1060 cm^{-1} is observed in the FTIR-ATR spectra (Grube *et al*., 2002).

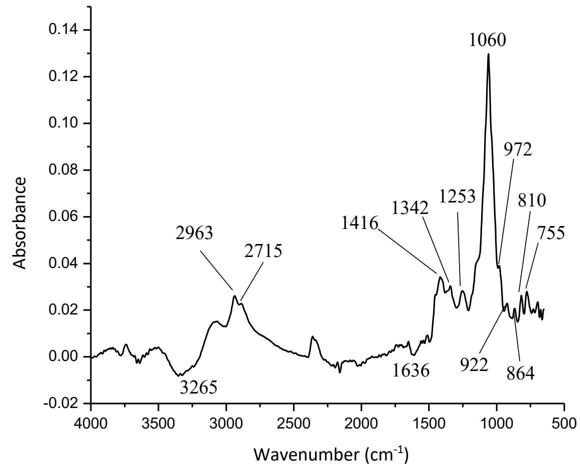

Figure 2. FTIR-ATR spectrum of agave juice at finish step of cooked (48 h) after water spectrum was subtracted. Spectrum in the interval between 4000 and 500 cm^{-1}.

The FTIR-ATR spectra of the agave heads juice after cooking (48 h) are shown in Figure 2. The peaks associated with the fructose molecule can be observed in the region between 1200 and 750 cm^{-1}, indicating relatively a complete transformation of a sample from juice rich in fructans to juice rich in fructose. Also, Figure 2 shows small peaks located at 972, 922, 864, 810, and 755 cm^{-1}, associated with a combination of vibrations of the C-C, C-O, and C-H bonds from the fructose molecule (Grube *et al*., 2002). Around 1060 cm^{-1} an intense peak was associated with a combination of stretching of the C-O and C-C bonds. The peak at 1253 cm^{-1} was related to C-O and C-C stretching. At 1342 cm^{-1} a peak associated with the O-H bending of the C-OH group appears. The peak observed around 1416 cm^{-1} corresponded with the combination of O-H bending and C-H bending of the alkenes. The peak located at 2963 cm^{-1} was assigned to the stretching of the C-H bond from the CH_2 group and at 2715 cm^{-1} there was a peak assigned to the C-H bond from the CH_3 group. As shown in Figure 2, two negative peaks are located approximately at 3265 and 1636 cm^{-1}, which come from the O-H stretching and H-O-H bending from the water molecule, respectively. The negative peaks were due to lower water concentration in the cooked juice compared to the reference used (100% water), as a consequence of the spectral subtraction: juice-water.

On the other hand, the evolution of the FTIR-ATR spectrum during the cooking process is shown in Figure 3, where the gradual disappearance of the peaks coming from the raw agave juices was observed,

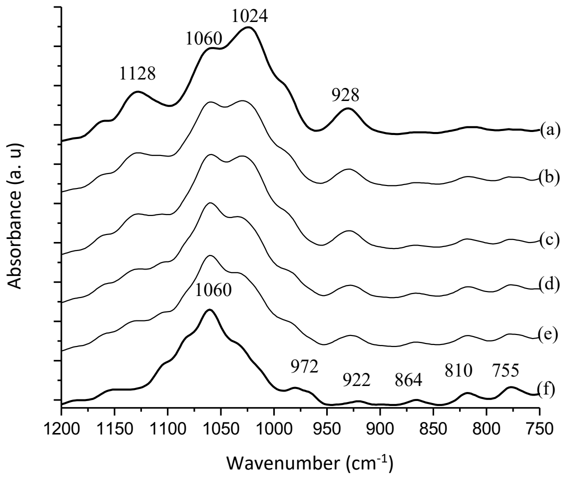

Figure 3. Transformation of the FTIR-ATR spectrum in the interval between 1200 and 750 cm^{-1} of the samples since juice uncooked (rich in agave fructans) until final transformation in fructose, during different cooking times at 95 °C: (a) 0 h, (b) 6 h, (c) 12 h, (d) 24 h, (e) 36 h and (f) 48 h.

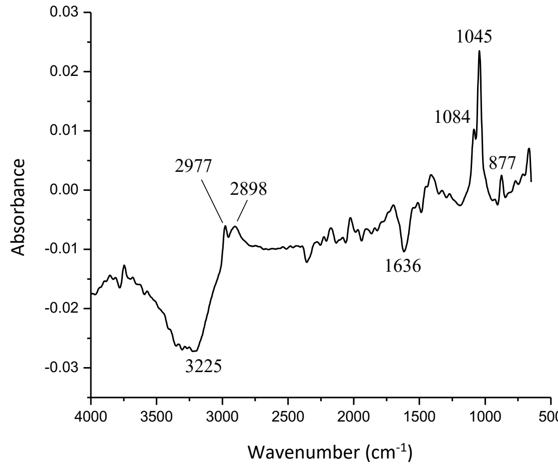

Figure 4. FTIR-ATR spectrum of agave juice fermented in the finish step (48 h at 35 °C) after substrate water spectrum in the interval between 4000 and 500 cm^{-1}.

e.g. peaks located at 1128, 1024, and 928 cm^{-1} and the continuous formation or deconvolution of the observed peak at 1060 cm^{-1}, which it is characteristic in an FTIR spectrum of fructose (Cadet *et al*., 1997). The FTIR spectrum of the final cooking sample only showed peaks associated with the fructose molecule, including the peaks of lower intensity located at 972, 922, 864, 810, and 755 cm^{-1}, which can be inferred from a satisfactory cooking process of the agave heads. Therefore, efficient monitoring of the transformation of fructans to fructose can be carried out by FTIR-ATR in a fast, and accurate way at the pilot or industrial level.

3.3 *FTIR-ATR spectroscopy characterization at the fermentation stage*

In tequila production, the fermentation process is one of the most relevant stages. Fermentation determines the ethanol productivity and the formation of volatile compounds in an alcoholic beverage. The fermentation of the agave cooked juice was carried out by the yeast *Saccharomyces cerevisiae*. The fermentation kinetics depends on several factors, such as the chemical composition of the cooked agave juices, the operating conditions of the process, and particularly the yeast strain used. The monitoring of the fermentation process for obtaining ethanol in alcoholic beverages has been successfully analyzed using FTIR spectroscopy. For example, Di Egidio *et al*. (2010) monitored by FTIR-ATR the fermentation of fructose in ethanol using *Saccharomyces cerevisiae* as a culture medium to obtain red wine. Also, Wu *et al*. (2015) monitored during the fermentation of Chinese rice wine different parameters such as ethanol, sugars, and total acidity using FTIR-ATR spectroscopy.

Figure 4 depicts the FTIR-ATR spectrum showing the peaks associated with the final fermented sample. In this figure, the peaks related to the ethanol molecule can be observed in the range between 1200 and 650 cm^{-1}, which indicates a relatively complete transformation of the fructose into ethanol in the fermentation process. The peaks located at 1084 and 1045 cm^{-1} were due to stretching vibrations of the C-O bond (Zeinalipour-Yazdi and Loizidou, 2021). The peaks that appear at 2977 and 2898 cm^{-1} were associated with the stretching of the C-H bond. Around 3225 cm^{-1} a negative peak was located, which can be associated with the stretching of the O-H bond from the water molecule. Also, the negative peak located at 1636 cm^{-1} was associated with the water molecule. In other regions of the spectrum, peaks associated with minor compounds present in the fermented sample also can be observed, probably due to bonds associated with the biomass (dead yeast), non-fermentable sugars, or organic acids. Figure 5 shows the spectral transformation of fructose into ethanol during the fermentation process (0-48 h). In the FTIR-ATR spectrum of the initial sample (0 h) the peak associated with fructose, located around 1060 cm^{-1}, disappears gradually in the interval from

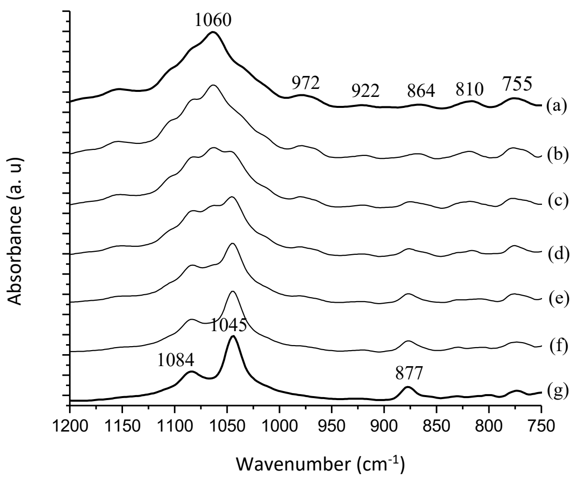

Figure 5. Transformation by fermentation of the FTIR-ATR spectrum in the interval between 1200 and 750 cm^{-1} of fructose (sample cooked at 48 h at 95 °C) to ethanol spectrum, during different fermentation times at 35°C: (a) 0 h, (b) 8 h, (c) 16 h, (d) 24 h, (e) 32 h, (f) 40 h and (g) 48 h.

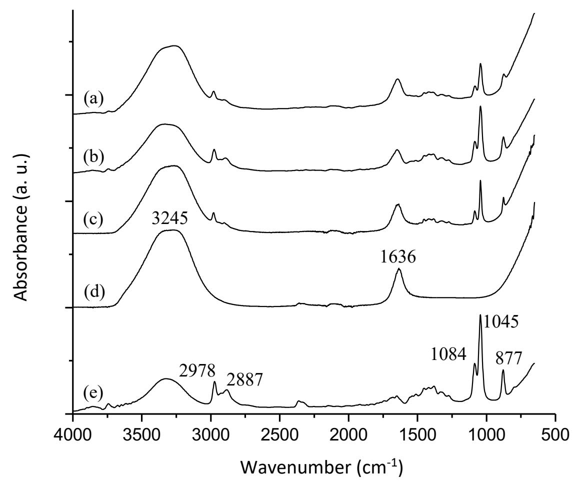

Figure 6. FTIR-ATR spectra of tequila from: (a) first distillation (20-25%), (b) second distillation (55-60%), (c) adjusted to commercial alcoholic grade (40% GL). (d) is the spectrum of water distillated and (e) is the spectrum of ethanol grade reactive (98% pure).

6 to 48 h. Afterward, in the FTIR spectrum of the final sample of the process, it can be observed the progressive formation of two peaks (at 1084 and 1045 cm^{-1}). As previously mentioned, these peaks were associated with the stretching vibration in the C-O bonds in the ethanol molecule. In addition, the peak that appears at 877 cm^{-1} in the final sample of the fermentation began its transformation even from the start of the fermentation process. Therefore, the fermentation stage can be followed in the range between 1200 and 700 cm^{-1} of the FTIR spectrum, in which the transformation of fructose peaks to ethanol could be detected.

3.4 *FTIR analysis of the tequila distillation process*

Distillation is a process in which ethanol and other compounds that give tequila its remarkable characteristics of aroma and flavor are concentrated. In the case of tequila, the ethanol obtained from fermentation, with content between 4 and 7% by volume, is subjected to a double distillation process. In the first distillation, the product has between 20 and 25% volume of ethanol. In the second distillation process, the ethanol content in the product is concentrated between 55 and 60% by volume. Subsequently, the distilled tequila is adjusted with water until reaching an ethanol concentration of around 40% by volume.

Traditionally the ethanol concentration in a distilled tequila is measured by densitometry and the result is expressed as a percentage of alcohol volume. In the FTIR-ATR spectra of the samples obtained from the first and second distillation (Figure 6a and 6b, respectively), it is observed that the peaks correspond to bending vibrations from water (3245 and 1636 cm^{-1}) and ethanol (1084 and 1045 cm^{-1}). This can be seen more clearly when these spectra are compared with the spectra of distilled water and ethanol (98% of purity) (Figure 6c and 6d, respectively). Also, the FTIR-ATR spectrum of distilled tequila adjusted at 40% ethanol is presented (Figure 6e). In the first distillation the peaks located at 1084 and 1045 cm^{-1} (bonds from C-O of the ethanol molecule) were less intense in comparison to these same peaks observed in the spectrum obtained from the second distillation. The difference in the height of the peaks in the FTIR-ATR spectra (absorbance) was due to the variation of the ethanol concentration in the distilled samples, that was, 0.14 and 0.29 at 1045 cm^{-1} in samples from the first (22% GL) and second (56% GL) distillation, respectively. In the adjusted tequila spectrum, the heights of the peaks (absorbance) associated with the ethanol molecule showed an intensity dependable on the adjusted ethanol content with distilled water, that was, 0.22 for tequila at 40% GL. Therefore, the FTIR-ATR spectroscopy could be a helpful option to measure the result of the distillation process through the ethanol content using the absorbance at 1045 cm^{-1}.

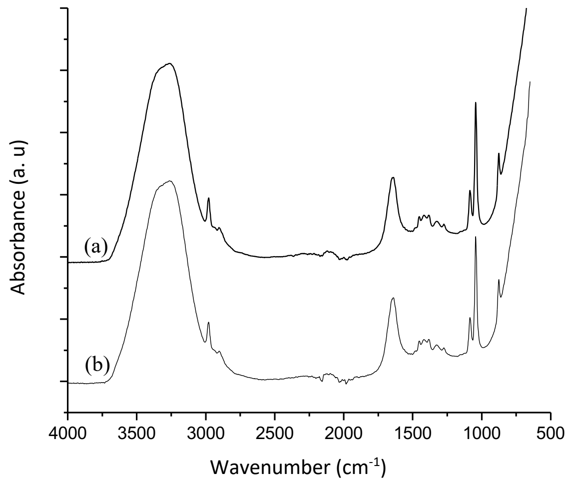

Figure 7. FTIR-ATR spectra of tequila blanco (a) and tequila reposado (b).

3.5 FTIR-ATR spectrum of tequila monitored during the aging process

The distilled or "*tequila blanco*" can be maturated by storing the tequila in oak barrels during a certain time. When the tequila is matured from 2 months and up to a year, it is known as "*tequila reposado*", while when the tequila remains in the barrel for at least one year it is called tequila aged or *tequila añejo*. The FTIR-ATR spectrum of an aged tequila does not present noticeable changes with respect to a FTIR-ATR spectrum of a *tequila blanco* (Figure 7). However, it can be observed that when the FTIR-ATR spectrum of the residue on the ATR crystal is obtained, as a consequence of the evaporation of the ethanol and the water contained in the sample of *tequila reposado*, a series of peaks was perfectly observed in the interval between 1300 and 900 cm^{-1}, Figure 8. The evaporation occurs in a short time of approximately 13 min on the ATR device, where first the ethanol was evaporated, and later the water (Figure 8). The origin of these peaks observed in the spectra come from compounds of the barrel originated from the interaction between the tequila and the walls of the oak barrel. The position shown of the peaks, it is likely that they come from bonds like: C-H, C-O, C-C or C-O-C, among others. These bonds could be related to organic molecules of the following families: acids, esters, and long-chain aldehydes, carbohydrates, furans, terpenes, phenols, etc. These compounds have been determined in tequila samples matured by other analysis techniques and their origin has been attributed to the interaction between the oak wall and the tequila (Muñoz-Muñoz *et al*., 2008).

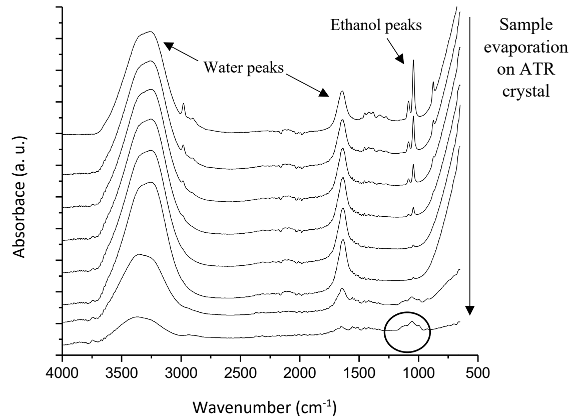

Figure 8. Sequence of peaks transformation of FTIR-ATR spectra for sample of tequila reposado evaporated on ATR crystal. It can be observed that first disappear peaks of ethanol and following peaks of water. The final FTIR-ATR spectrum showed peaks in the interval between 1300 and 900 cm^{-1} associated with molecules of compounds from oak barrel.

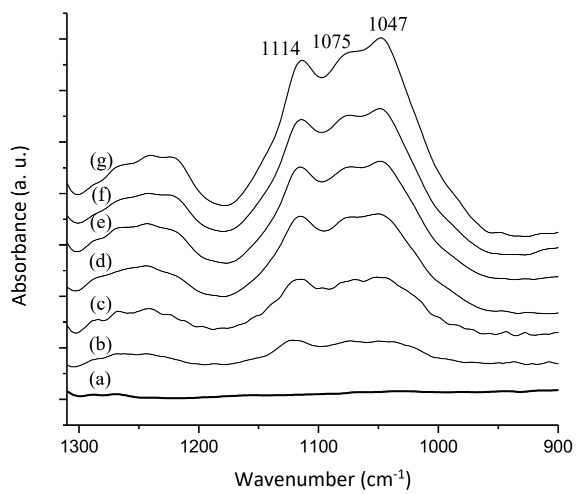

Figure 9. FTIR-ATR spectra in the interval between 1300 and 900 cm^{-1} that showed the transformation of the peaks since tequila blanco and during different times of aged in oak barrel: (a) tequila blanco, (b) 10, (c) 20, (d) 30, (e) 40, (f) 50 and (g) 60 aged days.

On the other hand, Figure 9 shows the evolution of the FTIR-ATR spectra in the interval between 1300 and 900 cm^{-1}. This measurement regards the residue left after evaporation of the ethanol and water, on the ATR crystal of samples obtained at different times of maturation. In this figure, it is possible to observe the formation of peaks after 10 days of maturation. These peaks gradually increase until the end of maturation (60 days), as can be seen in the FTIR-ATR spectrum (Figure 9-g). In this spectrum appear two perfectly distinguishable peaks: at approximately 1114 cm^{-1} and another at 1047 cm^{-1}. Also, one shoulder can be found at approximately 1075 cm^{-1}. These peaks were in the spectral region known as a fingerprint; therefore, its origin comes from a series of vibrations of different molecular bonds. This spectral variation observed during the aging of "*tequila blanco*" could help at an industrial level to carry out an adequate control in a fast and effective way of the aging process behavior in white oak barrels.

Conclusions

FTIR-ATR spectroscopy is a technique that can be used to monitor in a rapid and reliable way the different stages of the tequila production process. The raw juices spectra showed peaks associated with the water and agave fructans. The spectra obtained from samples of cooked juices at different times at 95°C showed the transformation of agave fructans to fructose. The spectra of the fermentation samples showed a sequence of the gradual transformation of fructose to ethanol. In the distillation step, FTIR-ATR spectra showed that the peaks can be associated with the alcoholic degree of the beverages and can be an alternative to determine the alcoholic degree of tequila. In the aging stage, the degree of the aging was monitored during the maturation time in function of the changes observed in the FTIR-ATR. These spectra were obtained by evaporation of the sample placed on the ATR crystal in the interval between 1300 and 900 cm^{-1}, where the peaks observed could be associated with compounds from the burned oak barrel.

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