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FTIR ANALYSIS OF HYDROTREATED *Jatropha curcas* L. SEED OIL OVER Ni-Mo CATALYST FOR BIOFUEL PRODUCTION

ANÁLISIS FTIR DEL ACEITE HIDROTRATADO DE SEMILLAS DE *Jatropha curcas* L. SOBRE CATALIZADOR Ni-Mo PARA LA PRODUCCIÓN DE BIOCOMBUSTIBLES

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Abstract

The catalytic hydroconversion of vegetable oil (CHVO) process associate vegetable oil triglyceride transformation to linear-chain alkanes, via hydrodeoxygenation reactions (HDO) to generate multiple hydrocarbon compounds similar to heavy vacuum oil. The CHVO process was applied to *Jatropha curcas* seed oil, on commercial hydrodesulfurization (HDS) Ni-Mo/Al_2O_3 catalyst under HDS operation conditions. *Jatropha curcas* seed oil triglycerides conversion were observe by Fourier Transform Infrared Spectroscopy (FTIR) and validated by Gas Chromatography (CG/MSD). The process occurs through three reaction pathways including: decarbonylation, decarboxylation and hydrodeoxygenation where the carboxyl group is removed from the fatty acids to produce alkanes similar to fossil fuels. Infrared spectroscopy in CHVO process showed to be a useful method to evaluate the catalytic activity by monitoring specific frequencies intensities of C=O and C-O bonds in carboxylic group. The intensity reduction of these bonds to linear hydrocarbons it associated as a way to quantify for CHVO process.

Keywords: *Jatropha curcas* L, biofuels, hydrodesulfurization, hydroconversion.

Resumen

La hidroconversión catalítica de aceite vegetal (CHVO) es un proceso que asocia la transformación de aceite vegetal a alcanos de cadena lineal. El proceso ocurre a través de reacciones de hidrodesoxigenación (HDO) para generar múltiples compuestos similares a los hidrocarburos de gasóleo. El proceso CHVO se aplicó al aceite de semilla de *Jatropha curcas*, con un catalizador comercial de hidrodesulfuración (HDS) Ni-Mo/Al_2O_3 en condiciones de operación de HDS. La conversión de los triglicéridos presentes en aceite de semillas de *Jatropha curcas* fue observada por espectroscopía de infrarrojo (FTIR) y validado por cromatografía de gases (GC / MSD). El proceso se produce mediante tres vías de reacción que incluyen: descarbonilación, descarboxilación e hidrodesoxigenación donde el grupo carboxilo se elimina de los ácidos grasos para producir alcanos similares a los presentes en combustibles fósiles. La espectroscopía de infrarrojo en el proceso de hidroconversión catalítica de aceites vegetales (CHVO) mostró ser un método útil para evaluar la actividad catalítica mediante el control de la intensidad de frecuencias específicas de enlaces C = O y C-O. Al cuantificar la reducción de intensidad de estos enlaces, se asoció como una forma de reducción del grupo carboxílico hacia hidrocarburos lineales para el proceso CHVO.

Palabras clave: *Jatropha curcas* L, biocombustibles, hidrodesulfurización, hidroconversión.

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1 Introduction

Each year the demand for liquid fuels increases, resulting in an equivalent increase in environmental pollution due to their excessive use. However, due to a declining in the fossil fuel reserves and environmental pollution concerns, it is important to search for alternative sources of energy. Biofuels are a potential alternative for the future that would provide a major source of energy because the processes are economically sustainable and would ensure compliance with environmental regulations (Kubickova and Kubicka, 2010).

Natural fats and vegetable oils are a potential feedstock for biofuel production. Currently, edible and non-edible vegetable oils are the main feedstock for biodiesel production M.M. González-Brambila, *et al*., 2014). These oils, consists of fatty acid methyl esters (FAMEs) that are produced by a chemical transesterification process of triglycerides with methanol or ethanol (Demirbas, 2003) or ultrasonic process (Soto-León *et al*., 2014). However, the FAMEs can develop undesirable properties derived from natural oil including low cetane number, high viscosity and low flow properties that are determined by the source oil composition (Maher and Bressler, 2007). These are increasing requirements on the feedstock quality resulting in higher prices.

A novel alternative for biofuel production is the catalytic hydrodeoxygenation and hydrocracking of vegetable oil feedstocks (Kubickova and Kubicka, 2010; Kubicka *et al*., 2009). These processes occur in the presence of hydrogen at high pressure and relatively high temperatures. In addition, the quality of the fuels depends on of the catalyst activity and operating conditions. Therefore, the catalytic hydroprocessing of vegetable oils (CHVO) is a promising alternative for biofuel production (Fig. 1) (Hancsók *et al*., 2007), which could employ the existing infrastructure of petroleum refineries (Huber, 2007).

Conventional hydrotreating involves removal of heteroatoms, such as sulfur and nitrogen. The hydrocracking process involves saturation and breakage of C-C bonds to produce high quality gasoline and diesel fuels (Scherzer and Gruia, 1996). The CHVO product process has better flow properties compared to biodiesel by transesterification (Soveran, 1992).

Vegetable oil hydrotreating and its mixtures with heavy vacuum gas oil have been explored for biofuel production by, employing hydrotreating catalysts based on Ni-Mo/Al_2O_3 and Co-Mo/Al_2O_3 (Liu, 2011) under hydrodesulfurization (HDS) nominal operating conventional conditions (Bezergianni and Kalogianni, 2009; Bezergianni *et al*., 2010). Several hydrotreated vegetable oils have been studied including sunflower (Huber *et al*., 2007), cottonseed (Sebos *et al*., 2009) palm oil (Taufiqurrahmi *et al*., 2010) *Jatropha curcas* L oil (Murata *et al*., 2010) Jatropha cinerea oil (Soto-León., *et al*., 2014) residual oils (Santos *et al*., 2010) and soy lecithin (Díaz, and Sánchez-Castillo, 2016). The use of these raw materials depends on the availability, cost and the environment in each country.

Jatropha curcas L. plant is resistant to adverse environmental conditions and easily adapt. Therefore, this plant could be cultivated in more extended areas without competing for arable land. However, this is a toxic plant, but in some local varieties in Mexico have low concentrations of toxic compounds and antinutritional agents, such phorbol esters, which allows the generation of by-products in the extraction process as a protein-rich paste for use in animal feedstocks as well for human consumption (Martinez-Ayala, 2002). Therefore, due to the physicochemical properties of the seed oil (Shah *et al*., 2004; Sujatha *et al*., 2008) allow an easy feeding industrial CHVO processes (Pinto *et al*., 2005).

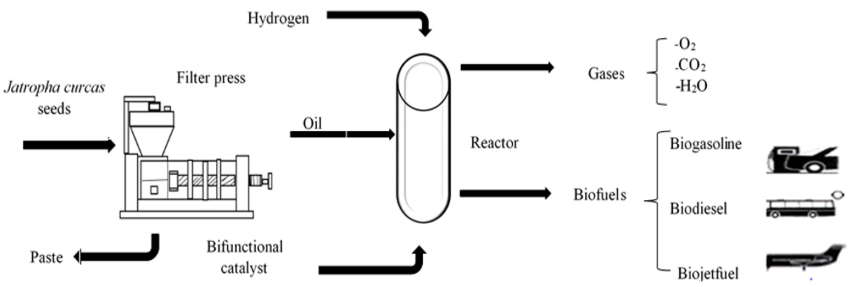

Fig. 1. Catalytic hydroconversion of vegetable oil (CHVO) process.

Gas chromatographic techniques are commonly used to determine selectivity and product yield. However, the gas chromatography (GC) method has some disadvantages like long analysis times; with higher operating costs compared to Fourier transform infrared spectroscopy (FTIR) technique. FTIR analysis method allows reliable readings in less time. Presenting the same sensitivity as the chromarography coupled with the test is nondestructive for the sample. For monitoring the CHVO process over *Jatropha curcas* seed oil FTIR technique permits to observe the change of fatty acids to linear hydrocarbons by the signal reduction of the carbonyl group.

2 Experimental

Jatropha curcas L seed species that were used in this study are native to Puebla state, Mexico. These varieties have a low phorbol esters content, therefore low toxicity. The seed oil was extracted by mechanical pressing using a mark Elvec 6 ton press capacity. The oil sulphur content is determine by ASTM-D4294 method using a SFLA-1100 h HORIBA equipment according to ISO8754 standards

Catalytic activity experiments were performed in a continuous flow tubular reactor using *Jatropha curcas* seed oil as the feedstock. Experiments were conducted under similar industrial HDS operation conditions with a temperature range of 310-390 °C, a pressure of 2.8 MPa, H_2/oil molar ratio of 15, and WHSV 176.4-35.28 h^{-1} (0.5 to .1 gr of catalyst). The catalyst bed used in this work was of Ni-Mo/Al_2O_3. Prior to the experiments, the catalyst was pre-sulfided in situ with a mixture of cyclohexane-CS_2 3% wt at 300 °C and 2.8 MPa with a flow of 0.1 ml/min for 4 hours for each experiment.

The infrared spectroscopy analysis was performed using a Bruker Infrared Spectrometer Vertex 70 with a resolution of 3 cm^{-1}, in the Attenuated Total Reflection (ATR) sampling mode. A diamond crystal with a single reflection was used and the transmission percentage values of this kind of plate are greater than 25. The measurement region was in the middle infrared, from 650 to 4000 cm^{-1}, sampling mode is equipped with the OPUS program for data acquisition.

Liquid products were analysed using a gas chromatograph GC/MSD Agilent 7890A, with autosampler G4513A coupled to a mass spectrometer 5975C VL MSD, with triple-Axis Detector, was used a capillary HP-5MS (30 m*320 μm *0.25 μm) column, with the external standard technique for quantification. The instrument control parameters were: oven program, 70 °C for 5 min then 20 °C/min to 200 °C for 5 min then 10 °C/min to 300 °C for 5 min, with an injection volume of 2 μl. With acquisition mode scan and temperatures of 230 °C and 150 °C for MS source and Quadrupole respectively.

A calibration curve was prepared to quantify the band intensity at 1743 cm^{-1} and 1160 cm^{-1} that represents the fatty acids abundance (Fig. 2). Using *Jatropha curcas* seed oil for the content of the maximum oxygenated compounds content and n-hexadecane as model hydrocarbon molecule as the minimum oxygenated compounds this compound was take an internal standard to represent the final products of HDO process. This analysis determines the fatty acid reduction to hydrocarbons by hydrodeoxygenation reactions over commercial Ni-Mo/Al_2O_3 HDS catalyst (Morgan *et al*., 2010). The signal reduction in the band intensity at 1743 cm^{-1} and 1160 cm^{-1} is quantified and correlated with the HDO conversion.

3 Results and discussion

Mechanically extracted *Jatropha curcas* seed oil is primarily composed of linoleic and oleic acids, as shown in Table 1. These data are consistent with those reported by Makkar *et al*., (2008) Becker and Makkar (2008). The seed oil sulphur content reaches to 80 ppm as glucosinolates types of sulphur compounds (El-Beltagi and Mohamed, 2010). These sulphur compounds were easily removed in the HDS process without adverse effects on process and catalyst (Hensen *et al*., 2003).

The liquid yield from the CHVO was 95%wt. The gas effluent content consisted primarily of CO_2, CO, H_2O, methane, and propane. The liquid effluent was analyzed by FTIR and the intensity variation in the absorption bands that are located at 3010 cm^{-1} (=C-H); 1743 cm^{-1} (C=O); and 1160 cm^{-1} (C-O) Fig. 3. These absorption bands are characteristics of the vibrational modes of carboxylic groups (Stuart, 2004), in figure 3 shows the changes in the intensity corresponding to the bands associated with the carboxyl groups reduction especially for the C=O and C-O bonds (Kirszensztejn *et al*., 2009). The signal reduction was quantified and correlated with the HDO conversion according to the calibration curve in figure 2 (Jitendra *et al*., 2011).

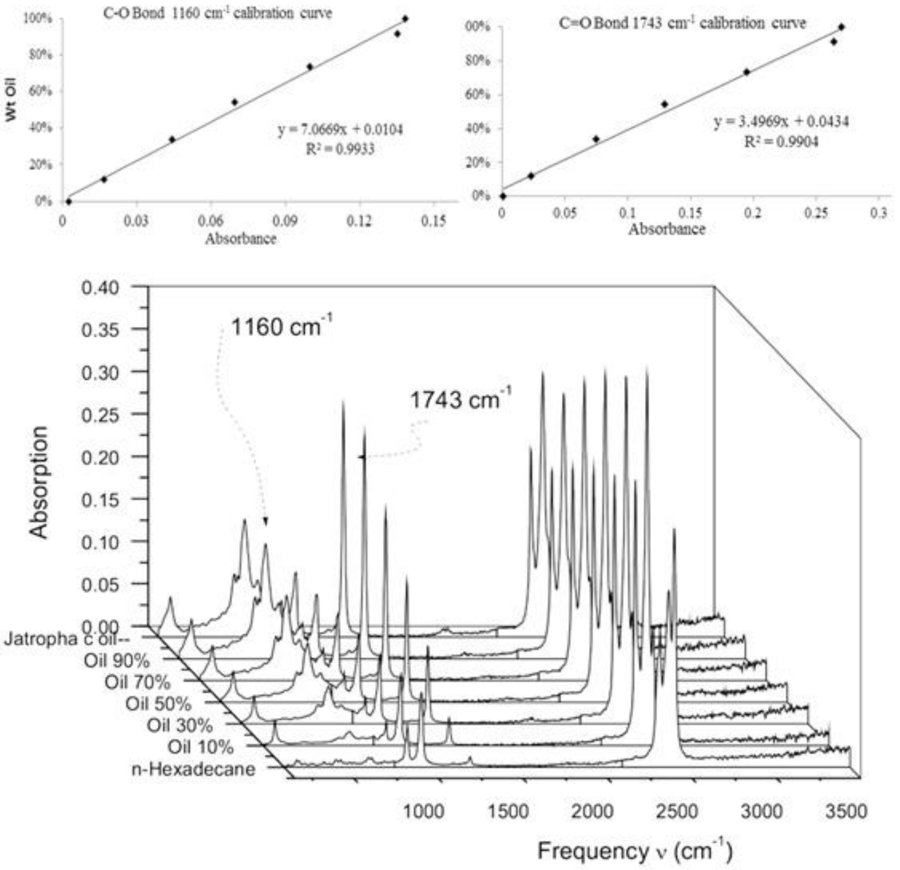

Fig. 2. FTIR Calibration curve of *Jatropha curcas* oil seed and n-hexadecane. Mixed in different proportions of oil 100% to 0%, hexadecane was taken as molecule model representing the full fatty acids deoxygenation process in the oil.

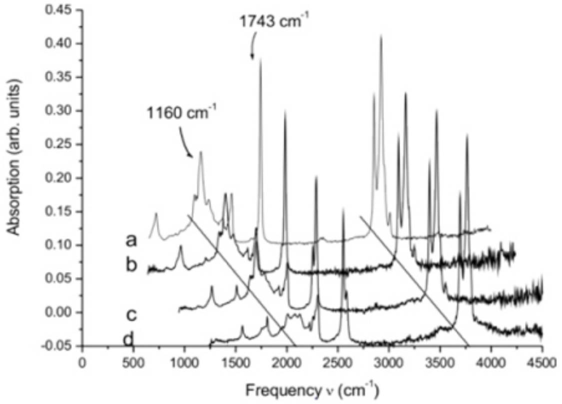

Fig. 3. Comparative FTIR spectra of hydrotreated *Jatropha curcas* oil with Ni-Mo catalyst, At 390°C, 2.8 MPa, WHSV=35.28 hr^{-1}; a) *Jatropha curcas* oil; b) Sample at 310°C; c) Sample at 350°C; d) Sample at 390°C.

Table 1. *Jatropha curcas* L. seed oil composition species native of state of Puebla.

Carboxylic acid	Composition (% wt)
Linoleic (18:2)	41.0-44.5
Oleic (18:1)	40.0-42.5
Palmitic (16:0)	9.5-11.0
Stearic (18:0)	2.0- 3.0
Myristic (14:0)	0.5- 1.0
Palmitoleic (16:1)	0.5 -1.0

Catalytic hydroconversion of *Jatropha curcas* seed oil depends on the catalyst used, the thermal cracking of triglyceride at 390 °C in non-catalytic processes are responsible for less than 5% of conversion. These data are consistent with those reported by Idem *et al.*, (1997) for canola oil, wherein the thermal cracking is significant above 400°C and increases with operating temperature. Therefore, the CHVO process is only performed by active catalyst sites. Commercial Ni-Mo HDS catalyst shows activity for CHVO process, increase conversion proportionality to temperature. At 310 °C, conversion levels reach of 15.6%, at 350 °C conversion level increase until 27.6% finally at 390°C conversion increase to 70.7% at WHSV 35.3 h^{-1}.

FTIR spectra of hydrotreated oil show a shift to lower frequencies in the vibrational change mode of the functional group present in fatty acids at 1743 cm^{-1} (Fig. 4). This phenomenon occurs in the presence of OH group (auxochrome) which alters the frequency and the intensity of the maximum absorption causing a displacement at 1710 cm^{-1} frequency, known as a bathochromic effect. These new structures have less stability than the corresponding carboxylic acids link by the absence of hydroxide group (Kubickova and Kubicka, 2010). These data suggest a gradual mechanism for decarbonylation, decarboxylation and hydrodeoxygenation reactions to form alkanes from fatty acids (Huber *et al.*, 2007) this sequential mechanism is recorded more clearly at the higher reaction temperature (Carlson *et al.*, 2008).

Catalytic activity is approximately three times higher at temperatures 390 °C such the conversion in CHVO process is favored with Ni-Mo dual-function catalyst. The catalyst improves molecular arrangement reactions attributed to acidity. Similar results has been reported by Chiappero *et al.*, (2010) and Jefferson *et al.*, (2006) who reported higher conversions of 80% and 90% respectively at higher WHSV values compared with those used in this research. However, the extent of these reactions depends on the catalyst type and hydroprocessing reaction conditions (Snare *et al.*, 2006). Formation of alcohols and esters has also been reported to take place during hydroprocessing of triglycerides (Guzman *et al.*, 2010). The use of sulfided catalysts during hydrodeoxygenation of triglycerides under HSD conditions produces carboxylic acids as intermediates (Vonghia *et al.*, 1995).

Analysis of the hydrotreated liquid products by GC/MSD was performed to validate FTIR technique (Fig. 5). The total triglycerides conversion was similar for both techniques, which has a 2.5% of error at 390 °C; 12.6% and 16 % of error at 350 °C and 310 °C respectively. The variance was attributed to the bathochromic effect in the FTIR analysis due to the presence of auxochromes (R-OH, R-COH), which alter the wavelength and absorption maximum intensity.

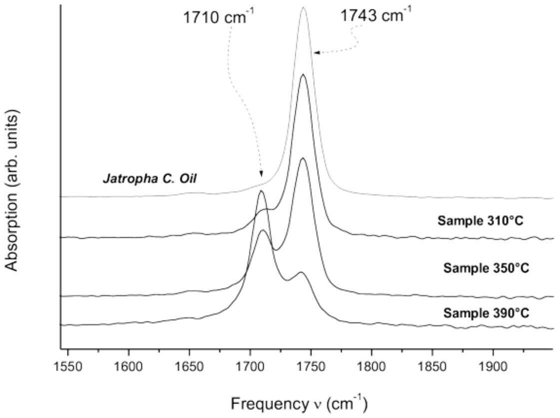

Fig. 4. Absorption band shift corresponding C=O bond at 1743 cm^{-1} with Ni-Mo based catalyst, at 2.8 MPa, WHSV= 35.28 hr^{-1}.

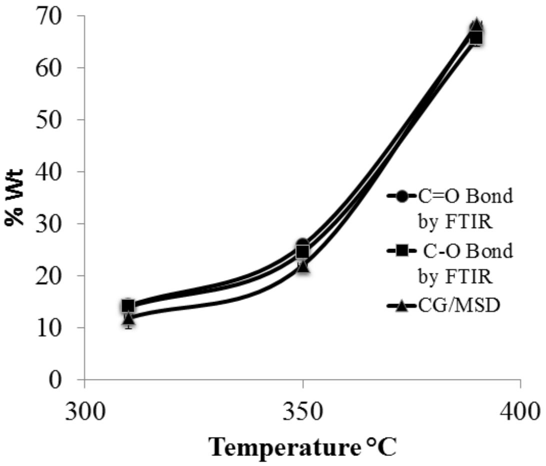

Fig. 5. Comparative conversion between FTIR and CG/MSD technique analysis with Ni-Mo based catalyst at WHSV= 35.28 hr^{-1} C=O bond by FTIR ●; C-O bond by FTIR ■; CG/MSD ▲.

The presence of other functional groups (i.e., ketones, aldehydes, and esters) at higher temperature makes the reaction tendency similar for both techniques. Five compound groups as including fatty acids, oxygenated, alkanes, alkenes, and aromatics in the liquid products were analysed by GC/MSD. For quantification was applied the external standard technique based on chain length and functional groups (Fig. 6).

14-Hexadecenal, 15-Heptadecenal, Pentadecanal, 9, 17-Octadecadienal and 2-Heptadecanone were the major intermediate products that constitute the 46% of the total effluent. Octene, 5-Undecene, 1-Hexadecene, 8-Heptadecene, 7-Octadecene, Nonane,

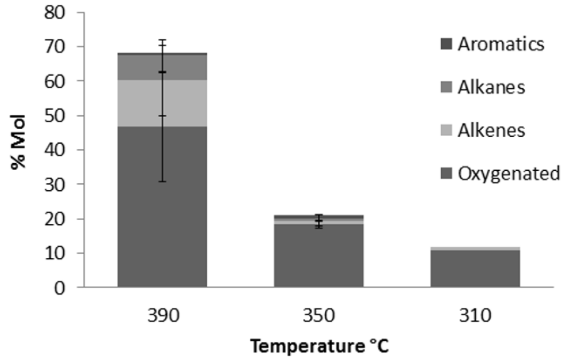

Fig. 6. Functional groups yield depending as a function of temperature for Ni-Mo based catalysts.

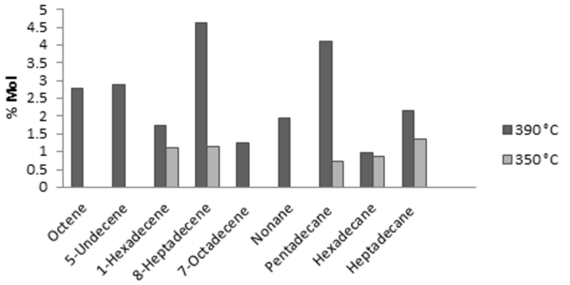

Fig. 7. Hydrocarbon formation basis for liquid biofuel production as a function of temperature for Ni-Mo based catalysts.

Pentadecane, Hexadecane and Heptadecane are the main products that constitute the 21% of the effluent as biohydrocarbon. Due to the feedstock is submitted to hydrotreating prior to hydrocracking so at a temperature of 310 °C hydrogenating/dehydrogenating activity is higher than acid activity. CG/MSD confirms the presence of intermediates oxygenated compounds in samples.

Hydrocarbons selectivity at the same temperature (i.e. alkenes and alkanes) increased as Weight hour space velocity (WHSV) increased of 0 to 21%. At temperatures of 310 °C there is no hydrocarbons presence by GC/MSD, only large amounts of oxygenates and free fatty acids compounds. Hydrocarbons selectivity increase up to 31% of product at 390 °C. The triglycerides degradation only by pressure and temperature effect is slight; the hydroconversion process to hydrocarbons formation is due to catalyst effect. Lui *et al*., (2011, 2012) used NiMo/Al_2O_3-SiO_2 catalyst to hydrogenate Jatropha seed oil to produce C_{15}-C_{18} n-paraffins, which were further cracked to generate C_{15}-C_{18} iso-paraffins. With a yield of 83.5% at a pressure of 4 Mpa, H_2/oil ratio 800 Nm^3/m^3 and Liquid hour apace velocity (LHSV) 7.6 h^{-1} these parameters are higher values compared with those used in this research. H_2/oil ratio 400 Nm^3/m^3 pressure of 2.8 Mpa. Murata *et al*., (2010) report conversion of 83.8% and yield of hydrocarbons of 67.7 % with similar conditions only with increased pressure of 6.5 Mpa.

Increasing the temperature in the CHVO process of 350 °C to 390 °C favors the formation of short chain hydrocarbon due to cracking effect of the original fatty acids. These compounds have the same number of carbon atoms in the molecule as the corresponding fatty acid chain, from which it was derived. However, these products have one less carbon atom than the original fatty acid chain (Fig. 7). The presence of heptadecane and pentadecane do suggest that decarboxylation also occurs.

Infrared spectroscopic analysis of the CHVO was used to evaluate the hydrodeoxygenation degree by monitoring the intensities of specific frequencies associated with carboxylic groups bond, and quantify the reduction transition in the HDS process. This technique does not destroy the sample and allows a, rapid and easy analysis. Based on the hydroconversion results the HDS Ni-Mo/Al_2O_3 catalyst is capable to transform the triglycerides found in *Jatropha curcas* seed oil to hydrocarbons as the basis for the biofuel production.

Conclusions

FTIR spectroscopic was used to analyze the hydroconversion process of *Jatropha curcas* seed oil. In this study, the peaks intensity at 1743 and 1160 cm^{-1} corresponding to the C=O and C-O bonds were monitored, correlated and quantified to the HDO conversion. High conversion of 68% was observed, this results are consistent with those obtained by CG/MSD.

In the hydroconversion process the catalyst, require greater WHSV values than those used in this work. These values were setting with the intention to observe functional groups deoxygenation changes in the oil, because the hydroconversion reactions of fatty acids are performed via a sequential mechanism that generates oxygenated intermediates. Therefore, the use of *Jatropha curcas* seed oil in the HDS process is a viable option for producing renewable diesel when the refining process is performed complete.

Mass spectroscopy analyzes indicate the presence in primary stages of oxygenates compounds such as aldehydes and ketones. These compounds appear as intermediates in early stages, as the process progresses the compounds change to alkenes and alkanes, as the main constituents of the final product. The reaction routes developed under the process conditions involve carbonylation and carboxylation due to the characteristics of the HDS catalyst. The thermal degradation is not significant under the studied conditions.

Nomenclature

MPa	operating pressure tubular reactor
Ni-Mo/Al_2O_3	nickel-molybdenum catalyst on an alumina support
WHSV	weight hour space velocity hr^{-1}
GC/MSD	gas chromatography coupled to mass spectroscopy detector
FTIR	Fourier Transform Infrared spectroscopy
Nm^3/m^3	normal cubic meters of gas/cubic meters of fluid

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