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From seeds to bioenergy: a conversion path for the valorization of castor and jatropha seeds

D.G. Montes-Núñez[a], G. Montero-Alpírez[a,✉], M.A. Coronado-Ortega[a], J.R. Ayala-Bautista[a], J.A. León-Valdez[a], A.M. Vázquez-Espinoza[b], R. Torres-Ramos[c] and C. García-González[a]

[a]Universidad Autónoma de Baja California, Instituto de Ingeniería, Mexicali, B.C., México.

[b]Universidad Autónoma de Baja California, Facultad de Ingeniería y Negocios, Mexicali, B.C., México.

[c]Universidad Autónoma de Baja California, Instituto de Ciencias Agrícolas, Mexicali, B.C., México.
✉Corresponding author: gmontero@uabc.edu.mx

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SUMMARY: The world's energy matrix can be diversified with biodiesel from castor and jatropha oil. Hence, the objective of this study was to assess a conversion path for the valorization of castor and jatropha seeds. The results showed the maximum extraction of castor oil at 90 °C, 2 rpm, and 6 mm nozzle, achieving a yield of 36.97% and for jatropha oil at 100 °C, 1.5 rpm, and 10 mm nozzle, achieving a yield of 20.11%. The acid value and cloud point of castor and jatropha oil were 0.797 and 23.44 mg KOH/g, 10±1 °C and 12±0.55 °C, respectively; while the pour point was -3 °C for both. The acid value and cloud point for biodiesels ranged from 0.26–0.43 mg KOH/g, and -12.50–6.10 °C, respectively. The viscosity of oils and biodiesel ranged from 0.02–1.3 P. GC-MS indicated 66.38% of methyl ricinoleate in castor biodiesel and 31.64% of methyl oleate in jatropha biodiesel. The HHV for castor and jatropha biodiesel ranged from 32.37–40.25 MJ/kg.

KEYWORDS: *Biodiesel; Castor; Jatropha; Seeds; Valorization.*

RESUMEN: ***De semillas a bioenergía: un camino de conversión para la valorización de semillas de ricino y jatrofa.*** La matriz energética mundial puede diversificarse con biodiesel de ricino y de jatrofa. Por lo tanto, el objetivo de este trabajo fue evaluar la ruta de conversión de las semillas de ricino y jatrofa. Los resultados mostraron que la máxima extracción de aceite de ricino se dio a 90 °C, 2 rpm, y boquilla de 6 mm, alcanzando un rendimiento de 36,97% y para el aceite de jatrofa fue a 100 °C, 1,5 rpm, y boquilla de 10 mm, obteniendo un rendimiento de 20,11%. El índice de acidez y punto de nube del aceite de ricino y jatrofa fue de 0,797 y 23,44 mg de KOH/g, 10 ± 1 °C y 12 ± 0,55 °C, respectivamente, mientras que el punto de fluidez fue de -3 °C para ambos. El índice de acidez y el punto de nube del biodiésel de ricino y jatropha fueron 0,43 y 0,26 mg KOH/g, -12,50 °C y 6,10 °C, respectivamente. La viscosidad dinámica de los aceites y el biodiesel osciló entre 0,02 y 1,3 P. El análisis GC-MS indicó 66,38% de ricinoleato de metilo en biodiesel de higuerilla y 31,64% de oleato de metilo en biodiesel de jatrofa. El HHV para el biodiésel de ricino y jatrofa osciló entre 32,37 y 40,25 MJ/kg.

PALABRAS CLAVE: *Biodiesel; Higuerilla; Jatropha; Semillas; Valorización.*

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1. INTRODUCTION

The international transport industry is the biggest energy consumer. According to the International Energy Agency (IEA), in 2018, energy consumption was 416.07 EJ, with crude oil being the primary source of energy at 41%. This impact prioritizes focusing efforts on developing new technologies to improve the energy efficiency of vehicles and the diversification of energy sources (Gay y García *et al*., 2014; Álvarez *et al*., 2017).

In 2019, the production of biofuels reached 163 million·m^3 internationally, where 69% corresponded to bioethanol production, and 31% was related to biodiesel production. The raw materials used in bioethanol production were corn with at a proportion of 66%, and sugar cane with 23%, while the remaining 11% came from sugar beet, cassava, among others. For biodiesel production, vegetable oils from palm, soybean, and rape seeds were used, at a proportion of 29, 25, and 17%, respectively, and the remaining 29% corresponded to used vegetable oils, animal fats, and virgin vegetable oils (Torroba, 2020).

The bioenergy potential in Mexico has been estimated between 3,000 to 3,459 PJ per year. This potential was determined without using land dedicated to food or protected natural areas (García *et al*., 2016). Despite the bioenergetic potential, in Mexico, only firewood and bagasse are considered primary biomass energy sources, which in 2019 contributed 247.92 and 113.25 PJ to primary energy production, respectively (SENER, 2019).

In search of new energy renewable sources, one option is oilseed crops, which is why Mexico has promoted planting these bioenergy crops. Among these crops, castor (*Ricinus communis*) and jatropha (*Jatropha curcas L.*) can be found as the major groups of crops with a production increase, research, testing, and most marketed globally. This is mainly due to their high oil contents (Comité Nacional Sistema-Producto Oleaginosas, 2005).

In 2019, 304.5 ha were reported to be planted with castor and 326 ha with jatropha (SIAP-SIACON, 2019). According to SAGARPA, in Mexico, there is a planting potential of 2,174,368 ha for castor and 1,914,853 ha for jatropha (SAGARPA, 2017).

Castor belongs to the Euphorbiaceae family and is also known as castor bean, mamona, ma hong liang, wonder tree, and castor oil plant. The castor bean is typical of semi-arid regions, and due to its adaptability, is currently cultivated in tropical, and subtropical regions (Chidambaranathan *et al.,* 2020). The jatropha belongs to the Euphorbiaceae family with approximately 175-200 different varieties (Piloto *et al.,* 2011). It is a succulent perennial shrub or small tree and can reach a height of 5 m or more, depending on conditions, such as type of soil, geographical location, and weather conditions. It is a drought and extreme (cold and heat) temperature-resistant plant.

Castor oil is extracted from its seeds, with approximately 45 to 50% (Chidambaranathan *et al.,* 2020); while the oil content in jatropha seeds ranges from 28 to 42% (Patel *et al.,* 2016). Castor oil comprises 90% ricinoleic acid, 4% linoleic acid, 3% oleic acid, 1% stearic acid, and less than 1% linolenic acid (Patel *et al.,* 2016). The composition of jatropha oil is generally 43.34% linoleic acid, 35.38% oleic acid, 15.32% palmitic acid, 4.06% stearic acid, and less than 2% palmitoleic, and linolenic acids (Okullo *et al.,* 2012). Due to the high ricinoleic acid content in castor oil, it is often used as biofuels, polymeric materials, drug, cosmetics, lubricants, and others (Patel *et al.,* 2016). Jatropha oil is used for biofuel (biodiesel) production, bio-lubricant, binderless particle board, pulping, paper, medicinal, and cosmetic uses (Ahmad *et al.,* 2016).

Biodiesel is one of the most widely produced biofuels, with an approximate production of 43,138 million liters worldwide (OECD-FAO, 2020). It is made by the reaction of triglycerides to short-chain alcohols using acid, alkaline, or enzymatic catalyst to obtain the mixture of fatty acid methyl esters known as biodiesel. It can be derived from various raw materials such as edible or inedible vegetable oils, animal fats, algal oils, and waste oils (Pradhan *et al*., 2014; Chidambaranathan *et al.,* 2020).

On the other hand, more than 95% of the world's biodiesel production has been made from edible vegetable oils (Chidambaranathan *et al.,* 2020). Rapeseed, soybean, sunflower, coconut, and palm oils have been the main raw materials for the production of biodiesel (Okullo *et al.,* 2012).

Hence, the objective of the present work was to assess a conversion path for the valorization of castor and jatropha seeds. The operating conditions to achieve the highest yield of castor and jatropha oil were obtained by mechanical extraction applying an experimental

design. The physicochemical properties of the oil and biodiesel from castor and jatropha according to international standards, GC-MS Chromatography, and the estimation of HHV were determined. The importance of this work is evaluating castor and jatropha crops as raw materials in the production of biofuels, highlighting that they do not compete with food, so they do not put the country's food security and sovereignty at risk. It will contribute to the diversification of the energy matrix at the national and international level, achieving a reduction in fossil fuel dependence.

2. MATERIALS AND METHODS

2.1. Moisture content

Crucibles at a constant weight with a 5 g sample of castor and jatropha seeds were placed in a Lindberg-Blue M oven at 105 °C to determine the moisture content of castor and jatropha seeds. The samples were dried for 4 hours (Cornejo, 2012). The moisture content (% MC) was determined by equation 1.

$$\text{Equation 1} \quad \%MC = \left(\frac{M_i - M_f}{M_i}\right)(100)$$

Where: M_i is the initial mass of the wet sample (g), and M_f is the final mass of the dry sample (g).

2.2. Mechanical oil extraction and filtration

A KOMET CA59G mechanical press was used to extract oil from castor and jatropha seeds. The procedure´s conditions of the mechanic press allowed for adjustments in temperature, speed, and nozzle size. In this specific equipment, the extraction pressure or extraction force could not be modified. An experimental design was developed for each seed sample since the proper oil extraction conditions were unknown. A factorial design was selected; variables were chosen by considering the oil´s fluidity gained without generating obstruction in the nozzle cake outlet. The following parameters were chosen for castor seed: temperature of 70 °C and 90 °C, speed 1 and 2 rpm, nozzle of 6 mm and 8 mm. Each treatment was performed in duplicate. The parameters used in the factorial design for jatropha seeds were: temperature of 95 °C and 100 °C, speed of 1 and 1.5 rpm, nozzle of 8 mm and 10 mm. Castor oil has certain advantages over jatropha oil since it requires a lower temperature for extraction by mechanical pressing, which implies a lower energy demand. Due to the limited availability of jatropha seed, a test was carried out for each treatment.

The collected oil was left to settle for 24 hours, the necessary time for the seed cake to settle and separate from the oil. Subsequently, it was vacuum filtered for the removal of impurities. A Kitasato flask, Buchner funnel, vacuum pump, and Whatman #2 filter paper (8 µm) were used. The Jatropha oil filtration time was 30 min. Castor oil was centrifuged twice due to its high viscosity: 1) 4,700 rpm for 30 min and 2) 1,500 rpm for 15 min. The oil yield was calculated by equation 2 (Hernández and Mieres, 2002).

$$\text{Equation 2} \quad \%Oil\ yield = \left(\frac{M_o}{M_s}\right)(100)$$

Where M_o is the extracted oil mass (g) and M_s is the processed seed mass (g).

For castor oil, due to the presence of hydratable phospholipids, a degumming process was required. In this process the oil was heated from 50 to 70 °C using a VWR hotplate/stirrer at 250 rpm. Distilled water was added at 3% mass based on oil weight, and the mixture was left under constant stirring for 30 min, while monitoring its temperature (Hernández and Mieres, 2002).

It was necessary to ensure the elimination of impurities to carry out an adequate characterization of the oils. Therefore, a vacuum filtration was done for both oils. The percentage of impurities removed from castor oil by vacuum filtration was lower than that of jatropha oil because its high viscosity made it difficult for the oil to flow in the filtration process. For that reason, it was necessary to subject the castor oil to a centrifugation process where 10.6% of impurities were removed from the oil in the first run and 2.23% in the second one.

2.3. Castor and jatropha oil transesterification

Methanol (CH_3OH) was used for both oils, considering an oil/alcohol molar ratio of 1:3, with 31% excess CH_3OH in volume, 1% sodium hydroxide (NaOH) as the catalyst and reaction temperature of 60 °C. The reaction time for castor oil was 30 min (Ferdous *et al.,* 2013), while for jatropha oil it was 120 min (Okullo *et*

al., 2012). 150 mL of previously degummed castor oil and 100 mL of jatropha oil were used for the reaction.

The obtained biodiesel was washed with a water-jet washing method to avoid emulsions. Distilled water was used and heated to 50 °C, the biodiesel: water ratio was 1:3. The number of washes was 5, determined by visual examination of the residual water. The biodiesel was dried at 110 °C for 10 min with constant stirring and stored in a dry environment.

2.4. Oil and biodiesel acid value

The acid value was determined according to ASTM D974, using potassium hydroxide (KOH), as shown in equation 3:

$$\text{Equation 3} \quad Av = \frac{56.1\,(N)(V)}{m}$$

Where *56.1*, *N,* and *V* are the molar mass (g/mol), normality, and the volume (mL) of KOH, respectively, and *m* is the mass of the sample (g).

Due to the high acid value present in jatropha oil, it was necessary to carry out an esterification process for 60 min, at 60 °C, and with constant stirring in a VWR hotplate/stirrer. For every 100 g of jatropha oil, 60 g of methanol (CH_3OH) were added. In addition, 0.27 mL of sulfuric acid (H_2SO_4) at 0.5% w/w were added as catalyst per 100 g of oil.

After the reaction, the mixture was transferred to a separatory funnel and allowed to settle for two hours. The resulting oil was dried with vigorous stirring at 110 °C for 10 min. The CH_3OH from the reaction was recovered by a rotary evaporator DLAB RE100-PRO at 55 °C and 160 rpm.

2.5. Cloud and pour point of oils and biodiesel

The cloud point was determined according to ASTM D2500. The pour point was determined according to ASTM D97. Both determinations were made in triplicate.

2.6. Dynamic viscosity

The dynamic viscosity was determined using a BROOKFIELD CAP 2000+ viscometer. The measurements were made in triplicate, with a temperature range of 50 to 100 °C. A #6 cone was used for castor oil, a #1 cone for jatropha oil, and a #2 cone for biodiesel.

2.7. Gas chromatography

Tests were carried out using an Agilent 7890A GC chromatograph attached to a 5975C mass detector, equipped with an HP-5MS capillary column (30 m x 0.25 mm x 0.25 µm). An automatic sampler was used to inject 1 µL of solution. The ionization energy was 70 eV with a mass range of 30 to 800 m/z. The initial temperature on the column was 125 °C for up to 0.5 min, the ramp set at 25 °C/min to 150 °C for up to 2 min, then to 200 °C with a speed of 50 °C/min. The injector temperature was set at 255 °C and the detector at 270 °C. The flow rate of the carrier gas (helium) was 1.0 mL/min injected with a 1:50 gas dilution. The identification of individual components was based on comparison with the NIST98 mass spectral library. All determinations were carried out in triplicate.

2.8. Higher heating value estimation of castor and jatropha biodiesel

The HHV for castor and jatropha biodiesel was estimated using the mass fraction and the molar fraction of the fatty acid methyl esters (FAME) present in the biodiesel. Equations 4 and 5 were used to calculate the HHV.

$$\text{Equation 4} \quad HHV = \sum_{i=1}^{n} (w_i)(HHV_i)$$

$$\text{Equation 5} \quad HHV = \sum_{i=1}^{n} (x_i)(HHV_i)$$

Where w_i, x_i, and HHV_i are the mass fraction, the molar fraction, and the higher heating value of a given FAME, respectively.

The mass fraction considered was obtained by gas chromatography, while the molar fraction was determined by direct conversion from its mass fraction. To estimate the HHV_i equation 6 was applied (Ramírez *et al.,* 2012):

$$\text{Equation 6} \quad HHV_i = 46.19 - \frac{1794}{M_i} - 0.21N$$

Where: M_i and N are the molecular weight and the number of double bonds in a given FAME, respectively. For this equation, an average abso-

lute deviation of 1.92% is reported between the experimental and calculated HHV's when using the mass and molar fractions. This equation was used to estimate the HHV of beef tallow oil, soybean oil, sunflower oil, corn oil, and cottonseed oil (Ramírez *et al.,* 2012). The estimated HHV was compared to the methodology proposed by Fassinou (2012).

3. RESULTS AND DISCUSSIONS

3.1. Moisture content

The reported moisture content of castor seeds varies from 1.84–5.4% (Omari *et al.,* 2015) compared to that obtained of 3.74%. For the jatropha seeds, the reported moisture content ranges between 4.75–19.57% (Garnayak *et al.,* 2008) compared to that obtained of 4.7%. Castor and jatropha seeds have a moisture content within the reported range, making them less susceptible to deterioration by microorganisms to be stored without affecting their viability.

3.2. Mechanical extraction of oils

Table 1 shows the ANOVA corresponding to the factorial design of castor and jatropha seeds, respectively.

It was concluded that the significant factors were B and C for castor oil and A and C for jatropha oil.

The Minitab® version 18 software was used for the analysis of the results, using residuals to verify the assumptions of normality, constant variance, and independence of the model, which are characteristics that corroborate the validity of the model, indicating that the response variable is normally distributed, with the same variance in each treatment and the measurements were independent. Equations 7 and 8 are the equations obtained from the experimental designs for castor (Y_C) and jatropha (Y_J) mechanical extraction, respectively.

$$\text{Equation 7} \quad Y_C = 179.8 + 9.97S - 9.29N - 0.6T + 0.37SN - 0.127ST + 0.0718NT$$

$$\text{Equation 8} \quad Y_J = -80.8 + 150.5S - 8.49N + 1.255T - 1.6SN - 1.14ST + 0.11NT$$

Where S, N, and T are the speed (rpm), nozzle size (mm), and temperature (°C), respectively.

Based on the statistical analysis, the optimal conditions for castor oil extraction were determined: 90 °C, 2 rpm, and a nozzle of 6 mm, achieving a yield of 36.97%. For the jatropha oil, the optimal conditions were: 100 °C, 1.5 rpm, and a nozzle of 10 mm, achieving a yield of 20.11%. The castor oil yield obtained was below the range of 38–48% by warm pressing (> 70 °C) reported by Scholz and Nogueira da Silva (2008), while jatropha oil was similar to the values achieved by other researchers where similar extraction equipment was used (Yate *et al.,* 2020).

Figure 1 illustrates the influence of significant factors on the performance of castor and jatropha oil extraction processes. In the case of castor seed, the maximum oil extraction was 110.9 g from a 300 g

Table 1. Castor and Jatropha oil ANOVA

Source	Castor oil[a]					Jatropha oil[b]				
	FD	SS	MS	FV	PV	FD	SS	MS	FV	PV
Speed (rpm) A	1	0.99	0.99	2.2	0.17	1	310.01	310.01	5061.3	0.009
Nozzle (mm) B	1	58.83	58.8	130.77	0	1	0.451	0.451	7.37	0.225
Temperature (°C) C	1	33.29	33.3	74.01	0	1	33.62	33.62	548.9	0.027
AB	1	0.55	0.55	1.22	0.3	1	1.28	1.28	20.9	0.137
AC	1	1.61	1.61	3.59	0.09	1	4.061	4.061	66.31	0.078
BC	1	2.06	2.06	4.58	0.06	1	0.605	0.605	9.88	0.196
Error	8	4.05	0.51	-	-	1	0.061	0.061	-	-
Total	15	101.4	-	-	-	7	350.08	-	-	-

[a]Performed in duplicate; [b]Performed in a single analysis; FD Freedom degree; SS Square sum; MS Mean square; FV F-Value; PV P-Value.

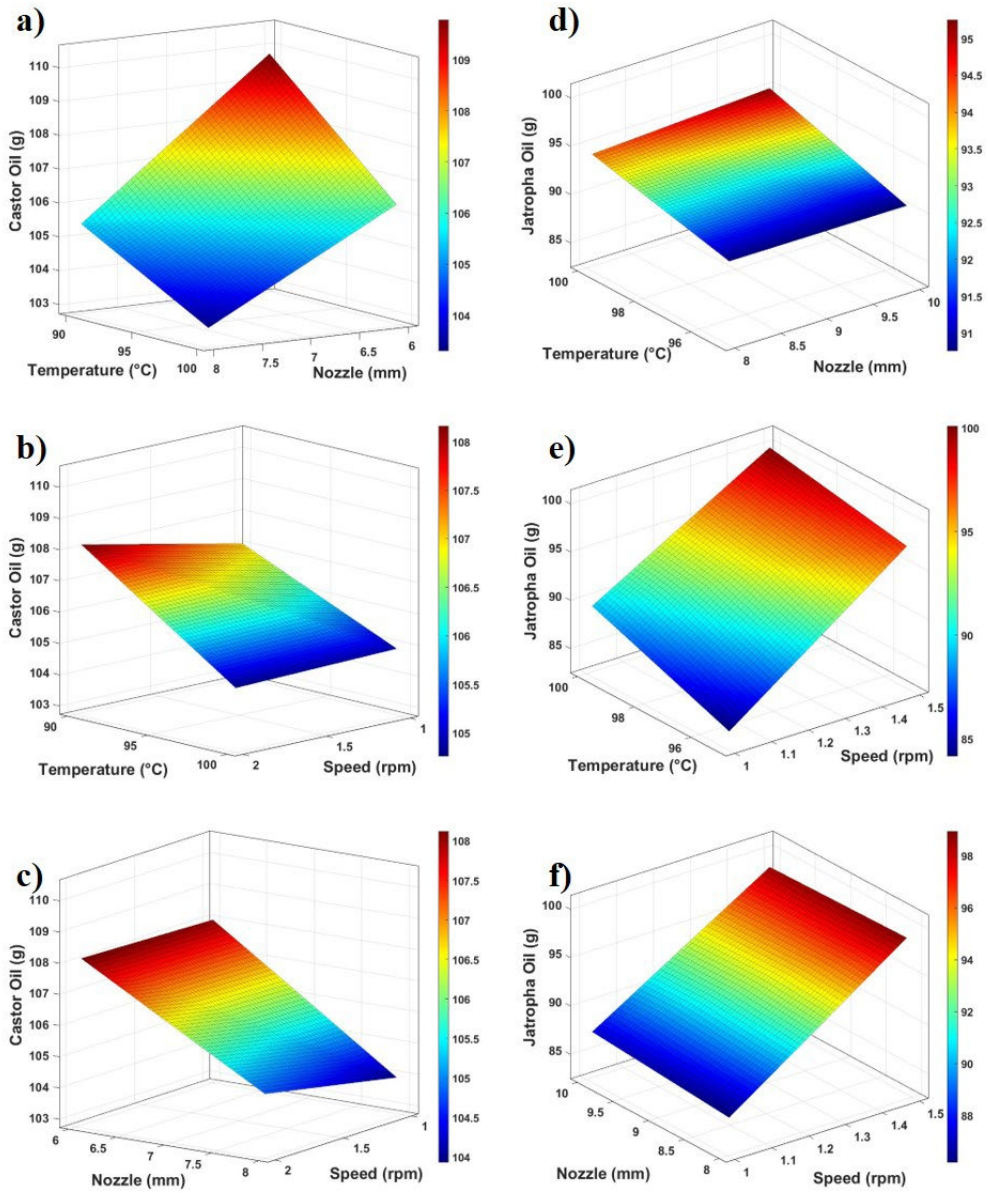

Figure 1. Response surface for castor and jatropha oil extraction. a) Temperature and nozzle vs castor oil, b) Temperature and speed vs castor oil, c) Nozzle and speed vs castor oil, d) Temperature and nozzle vs jatropha oil, e) Temperature and speed vs jatropha oil, f) Nozzle and speed vs jatropha oil. a), b) and c) performed in duplicate; d), e) and f) performed in a single analysis.

sample. For jatropha seed, the higher oil extraction was 100.55 g from a 500 g sample.

3.3. Transesterification of castor and jatropha oil

The conversion percentages of castor and jatropha oils to biodiesel were 96.66 and 98.88% respectively, which is a great advantage when used as raw material. The conversion percentage will depend mainly on the type and quality of the seeds, the pre-treatment, and the transesterification conditions applied. Other authors report conversion percentages for castor oil of 76% and jatropha oil of 91% (Okullo *et al.,* 2012). Likewise, castor oil requires a lower cost pre-treatment for its conversion into biodiesel.

3.4. Acid value, cloud point and pour point of oils and biodiesel

Table 2 displays the results of the analyzed properties for castor and jatropha oils, as well as the biodiesel obtained from them.

Table 2. Properties of castor oil, jatropha oil and biodiesel

Sample/Property	Acid value (mg KOH/g)[b]			Cloud point (°C)[c]				Pour point (°C)[c]			
	Present work	ASTM standard	Cited in Literature	Present work	(σ)	ASTM standard	Cited in Literature	Present work	(σ)	ASTM standard	Cited in Literature
Castor oil	0.797	2 (max)	0.44[d], 0.91[e]	10	-1	Report to customer	14[j]	-3	-1	Report to customer	-13[m], -15[n]
Jatropha oil	23.44 0.45[a]	2 (max)	0.1428[f], 0.7 to 1.7[g]	12	-0.6	Report to customer	14[f]	-3	-1	Report to customer	-3[o], -5[p], -6[p]
Castor biodiesel	0.43	0.5 (max)	0.25[h], 0.52[e]	-12.5	-1	-3 to -12	-18[k]	< -20	-	-15 to 16	-12[n], -30[k]
Jatropha biodiesel	0.26	0.5 (max)	0.18 to 0.29[i]	6.1	-0.6	-3 to -12	12[l]	0	-1	-15 to 16	-6 to 2[o]

[a]After esterification process; [b]Performed in a single analysis; [c]Performed in triplicate; [d]Omari *et al.*, 2015; [e]Pradhan *et al.*, 2012; [f]Ahmad *et al.*, 2016; [g]Piloto *et al.*, 2011; [h]Banerjee *et al.*, 2017; [i]Lu *et al.*, 2009; [j]Okullo *et al.*, 2012; [k]Chidambaranathan *et al.*, 2020; [l]Yate *et al.*, 2020; [m]Kumar *et al.*, 2020; [n]Tunio *et al.*, 2016; [o]Koh *et al.*, 2011; [p]De Oliveira *et al.*, 2009.

For biodiesel production, an acid value of less than 2 mg KOH/g is considered an essential condition for carrying out the transesterification reaction through alkaline catalysis, to avoid saponification (Mashad *et al.,* 2008). The acid values for castor and jatropha oils were within the ranges reported (Piloto *et al.,* 2011; Pradhan *et al.*, 2012; Omari *et al.,* 2015; Ahmad *et al.,* 2016); however, for jatropha oil, the acid value was higher than the value required to perform the transesterification reaction. Therefore, the percentage of free fatty acids was reduced by an esterification process using H_2SO_4 before transesterification (Mashad *et al.,* 2008). The high acid value for jatropha oil is attributed to the presence of oleic (C18:1) and linoleic (C18:2) acids since they are the two components with the highest proportion and present one and two unsaturations in their structure chemistry, respectively. Also, the high acid value for jatropha oil can be due to different factors such as the type of seed, the origin, and even the storage time (De Oliveira *et al*., 2009). After esterification, the free fatty acid content decreased by 98.13% at 60 °C, 60% w/w methanol, and 0.5% w/w of sulfuric acid, achieving an acid value of 0.45 mg KOH/g. The maximum acid value for biodiesel allowed by the ASTM D6751 standard is 0.50 mg KOH/g, so biodiesel from castor and jatropha oils is within the values reported in the literature (Lu *et al.,* 2009; Pradhan *et al.*, 2012; Banerjee *et al.,* 2017).

The cloud points measured for castor and jatropha oils are similar to those reported in the literature (Okullo *et al.,* 2012; Ahmad *et al.,* 2016). Castor biodiesel has a lower cloud point than jatropha biodiesel, making castor biodiesel more suitable for use in cold locations. Jatropha biodiesel may require the use of additives to improve its cloud point.

The pour points for castor and jatropha oil were reached at the same temperature, which was similar to those reported for jatropha oil (De Oliveira *et al.,* 2009; Koh *et al.,* 2011); while for castor oil, it ranges from -13 to -15 °C (Tunio *et al.,* 2016; Kumar *et al.,* 2020). However, it does not represent complications for its use in tropical areas and favors its ability to work at low temperatures. The measured pour points for jatropha oil biodiesel were within the reported ranges (Koh *et al.,* 2011). However, the pour point was not achieved for castor biodiesel due to equipment limitations, where the temperature reached was -20 °C, exceeding the measurement capacity. Temperatures of -12 to -30 °C are reported for the pour point of castor biodiesel (Tunio *et al.,* 2016; Chidambaranathan *et al.,* 2020). The difference between the values obtained and those reported in the literature may be due to factors such as the geographical area to which the seeds belong, climatic conditions, and the state of the crop.

3.5. Dynamic viscosity of oils and biodiesel

The viscosity of the oil used as raw material directly affects the viscosity of the biodiesel produced. Figure 2 exhibits the results of the tests to measure the viscosity of castor and jatropha oils and biodiesel.

In Figure 2 it can be seen that the viscosity of castor oil is much higher than that of jatropha oil. This is due to the hydroxyls present in the triglyceride molecule of castor oil, which give it this high viscosity character-

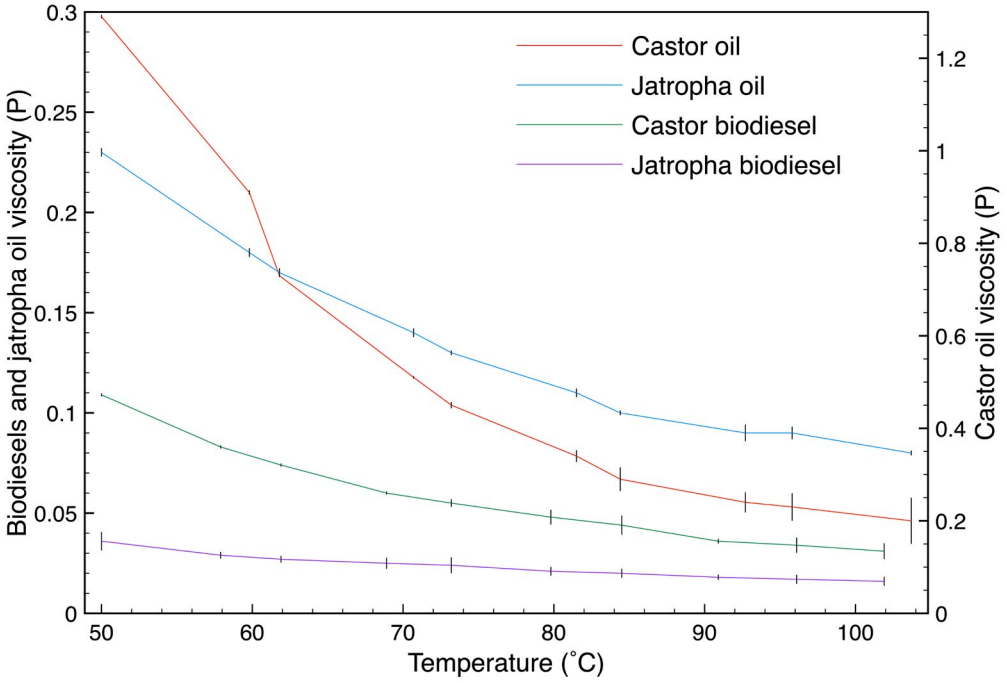

Figure 2. Dynamic viscosity at different temperatures, performed in triplicate. The standard deviation for castor oil ranges from 0.001 – 0.049 P; the standard deviation for jatropha oil ranges from 0.001 – 0.004 P; the standard deviation for castor biodiesel ranges from 0.0006 – 0.0046 P; the standard deviation for jatropha biodiesel ranges from 0.0012 – 0.0044 P.

istic. Viscosity increases with increasing chain length and decreases with the number of double bonds (level of unsaturation in the chain) (Silitonga *et al*., 2013). The presence of linoleic acid (C18:2) contributes to the decrease in the viscosity of jatropha oil since it represents the second compound with the highest proportion and presents two unsaturations in its chemical structure. The majority presence of ricinoleic acid (C18:1) in castor oil gives it a higher viscosity because this acid has one unsaturation in its chemical structure.

The viscosity of esters obtained from castor and jatropha oils through the transesterification reaction was significantly lower, achieving reductions of up to 85 and 80%, respectively, compared to the viscosity of the raw materials. Furthermore, castor biodiesel has a higher viscosity than jatropha biodiesel since castor oil has the highest viscosity of all known vegetable oils; however, its complete solubility in alcohol makes it suitable for conversion to biodiesel (Valderrama *et al.,* 1994).

3.6. Gas chromatography

The resulting chromatograms of biodiesel from castor and jatropha oil are exposed in Figures 3 and 4.

Table 3 displays the type and percentage of fatty acid methyl esters present in the extracted castor and jatropha oil.

Table 3. Methyl ester of fatty acid composition of castor and jatropha oil, expressed in percent of the total fatty acids

Component		Methyl Palmitoleate (C16:1)	Methyl Palmitate (C16:0)	Methyl Linoleate (C18:2)	Methyl Oleate (C18:1)	Methyl Elaidate (C18:1)	Methyl Stearate (C18:0)	Methyl Ricinoleate (C18:1)	Other components	ΣSFA[a]	ΣMUFA[b]	ΣPUFA[c]	TU[d]	TU/SFA index[e]
Castor oil	Mean[f]	-	6.4	7.54	13.79	1.2	3.4	66.38	1.29	9.8	81.37	7.54	88.9	9.07
	σ	-	0.006	0.008	0.01	0.002	0.003	0.024	-	-	-	-	-	-
Jatropha oil	Mean[f]	0.93	13.75	27.13	31.64	1.69	5.2	0.7	18.97	18.95	34.96	27.13	62.1	3.28
	σ	0.0003	0.0009	0.0006	0.0027	0.0033	0.0004	0.0029	-	-	-	-	-	-

[a]ΣSFA: saturated fatty acid; [b]ΣMUFA: monounsaturated fatty acid; [c]ΣPUFA: polyunsaturated fatty acid; [d]TU: total unsaturated fatty acid; [e]TU/SFA index: total unsaturated fatty acid/saturated fatty acid index; [f]Performed in triplicate.

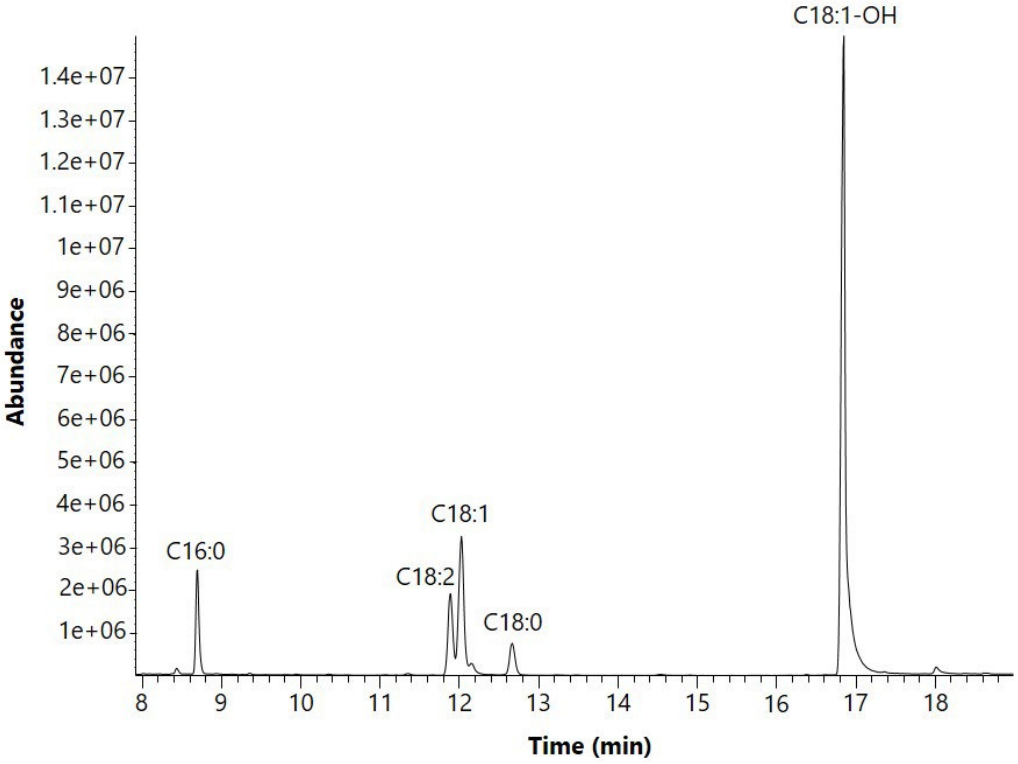

Figure 3. Chromatography of filtered castor oil.

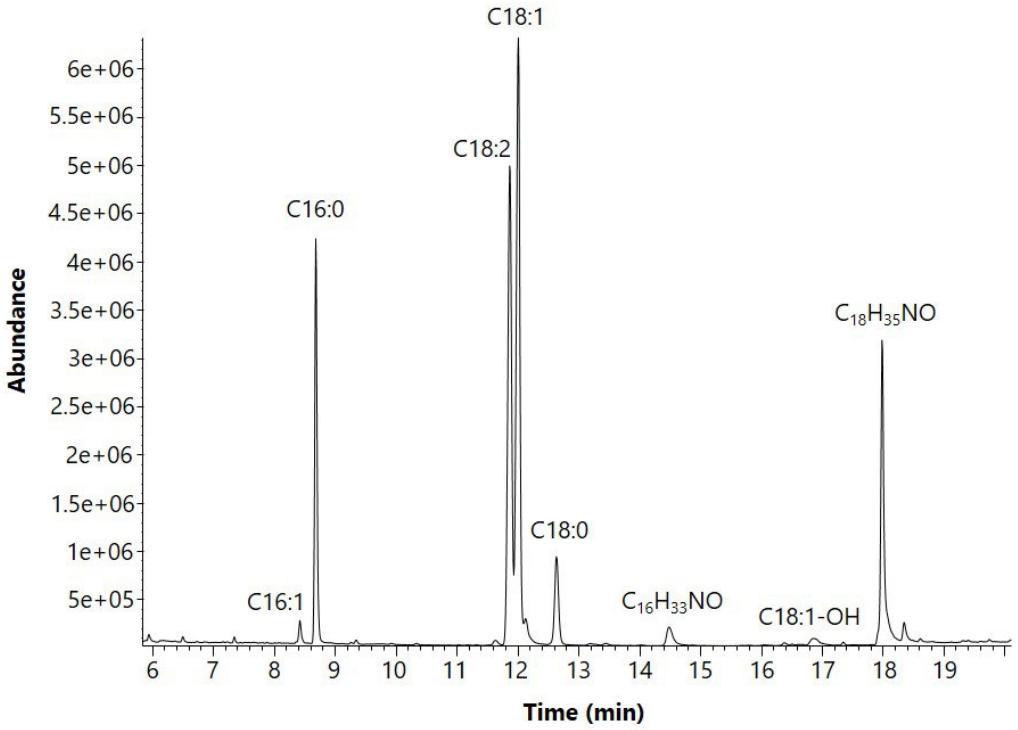

Figure 4. Chromatography of filtered jatropha oil.

The high presence of nitrogen compounds in biofuels is not desirable because when they burn, they become sources of NO_x in the exhaust gases (Kaewpengkrow *et al.,* 2013). Due to the chemical structure of FAME's, biodiesel can be more susceptible to oxidation than mineral diesel. Saturated FAME's increase cloud point, cetane number, and improve storage stability; while polyunsaturated FAME's decrease these properties (Das *et al.,* 2009). However, it has been reported that unsaturated FAME's increase fuel lubricity and improve cold flow properties (Hoekman *et al.,* 2012). Therefore, the presence of saturated and unsaturated FAME's is desirable for biodiesel production to obtain a fuel with better properties.

The central methyl esters in castor biodiesel were methyl ricinoleate (66.38%), methyl oleate (13.79%), and methyl linoleate (7.54%). These values were within the range of those in the literature (Okullo *et al.,* 2012).

The chromatographic profile of jatropha biodiesel indicates that the most representative compounds were methyl oleate (31.64%), methyl linoleate (27.13%), and methyl palmitate (13.75%), similar to literature values (Piloto *et al.,* 2011; Okullo *et al.,* 2012).

3.7. Higher heating value of castor and jatropha biodiesel

The HHV of biodiesel can be predicted by the FAME composition of biodiesel (Fassinou, 2012; Ramírez *et al.,* 2012). Table 4 reveals the values of w_i and x_i calculated with the relations taken from Table 3, and the HHV_i was calculated with equation 6.

Table 4 depicts that the compound with the highest contribution to HHV in castor biodiesel is methyl ricinoleate with 26.49–26.82 MJ/kg. The HHV reported for methyl ricinoleate is 40.37 MJ/kg (Fassinou, 2012), so comparing this value with that obtained in equation 6, there is a difference of 0.08%. For jatropha biodiesel, the two compounds with the highest contribution to HHV are methyl oleate with 12.68–15.36 MJ/kg and methyl linoleate with 10.81–13.18 MJ/kg. The reported values for the HHV of methyl oleate and methyl linoleate are 39.90 MJ/kg and 39.85 MJ/kg, respectively (Fassinou, 2012). Comparing these values with those obtained through equation 6 there is a difference of 0.45% for methyl oleate and 0.05% for methyl linoleate. Given the composition of the methyl esters, the HHV for castor biodiesel of 39.74 to 40.25 MJ/kg can be predicted; while for jatropha biodiesel, an HHV between the ranges of 32.37 to 39.94 MJ/kg can be expected. Ramírez *et al.,* (2012) report that the methodology for estimating the HHV of biodiesel should not be used for castor biodiesel, so it was compared to a methodology proposed by Fassinou, (2012). The difference between both methodologies was 0.27%.

When using blend biodiesel with diesel there is a decrease in HHV with an increasing mixing ratio. The HHV from B5 to B100 represents a range of 39.74 to 41.80 MJ/kg for castor biodiesel and 32.37 to 41.79 MJ/kg for jatropha biodiesel. The HHV of biodiesels from the current work are slightly lower than those of gasoline (40.43 MJ/kg), and diesel (41.40 MJ/kg) (SENER, 2019).

Table 4. HHV_i (MJ/kg), w_i and x_i calculated from FAME composition

FAME (Carbon chain)	M_i (g/mol)	HHV_i	Castor biodiesel		Jatropha biodiesel	
			w_i*HHV_i	x_i*HHV_i	w_i*HHV_i	x_i*HHV_i
Methyl Palmitate (C16:0)	270.46	39.72	2.54	2.91	5.46	7.25
Methyl Palmitoleate (C16:1)	268.44	39.46	-	-	0.37	0.49
Methyl Stearate (C18:0)	298.51	40.33	1.37	1.42	2.10	2.52
Methyl Oleate (C18:1)	296.50	40.08	5.53	5.77	12.68	15.36
Methyl Ricinoleate (C18:1)	313.50	40.40	26.82	26.49	0.28	0.32
Methyl Linoleate (C18:2)	294.48	39.83	3.00	3.16	10.81	13.18
Methyl Elaidate (C18:1)	296.50	40.08	0.48	0.50	0.68	0.82
Total	-	-	39.74	40.25	32.38	39.94

HHV_i Higher heating value of a given methyl ester; w_i Mass fraction of a given methyl ester; x_i Molar fraction of a given methyl ester; FAME Fatty Acid Methyl Ester; M_i Molar mass.

4. CONCLUSIONS

The physicochemical characterization of the seeds, oil, and biodiesel of castor and jatropha was carried out, and the determination of the necessary conditions for obtaining castor and jatropha oil by mechanical extraction. According to the experimental designs, the best extraction yields were at 90 °C, 2 rpm, and a nozzle of 6 mm for castor oil. For the jatropha oil, the optimal conditions were at 100 °C, 1.5 rpm, and a nozzle of 10 mm.

Castor seeds have a low energy requirement and show better performance in the oil extraction process, along with most of the properties determined for oil and biodiesel obtained from them. Castor oil presents better conditions without requiring high-cost pre-treatments, so it is more convenient to promote its use for the production of biodiesel. The conversion percentages of castor and jatropha oils to biodiesel were 96.66 and 98.88%, respectively.

Ricinoleic acid is responsible for the low acid value and high viscosity of castor oil, as it is the major compound in the oil.

Of all the measured parameters, the acid value turned out to have the most significant effect since the high acid value of the oil hinders the production and separation of methyl esters, which causes a decrease in the percentages of conversion to biodiesel, increasing losses and production costs. Linoleic acid is responsible for the high acid value and low viscosity of jatropha oil, as it is the second largest compound in the oil.

The heating value for biodiesel is closely linked to the properties of the most representative methyl esters, so it is to be expected that the heating value of biodiesel is similar or close to the heating value of esters. The HHV of castor biodiesel can be expected to be close to methyl ricinoleate HHV, as it is the major contributor with 66.38% (w/w). The HHV of jatropha biodiesel will be close to the average of the HHV of the most representative FAME.

Therefore, it is concluded that the methodology developed in the present work for castor and jatropha seeds allows for obtaining biodiesel which falls within the acceptance ranges of ASTM D6751 of the determined properties.

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