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Chapter 7

Biodiesel Current Technology: Ultrasonic Process a Realistic Industrial Application

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1. Introduction

Biodiesel is briefly defined as a renewable fuel derived from vegetable oils or animal fats. Similarly, the American Society of Testing and Materials (ASTM) defines biodiesel as monoalkyl long-chain fatty acids esters derived from fatty renewable inputs, such as vegetable oils or animal fats. The term "bio" refers to its origin from biomass related resources, in contrast to the traditional fosil-derived diesel, while the term "diesel" refers to its use on engines; as a fuel, biodiesel is typically used as a blend with regular diesel. To date, biodiesel is well recognized as the best fuel substitute in diesel engines because its raw materials are renewable, and it is biodegradable and more environmentally friendly; biodiesel probably has better efficiency than gasoline and exhibits great potential for compression-ignition engines.

Biodiesel was mainly produced from soybean, rapeseed and palm oils, although social and economic considerations have turned attention to second generation biomass raw materials such as *Jatropha curcas*oil [1]. It is well known that biodiesel competitiveness has to be improved, as to compare to curcas oil diesel, to spread out its consumption. Two routes are suggested to overcome this problem; one is related to get cheap raw materials (i.e., triglycerides, nonedible vegetable oils, animal fats and wasted oils), and other one is to reduce processing cost; notoriously both issues are interrelated [1]. The raw material origin is of great relevance because it determines the final biodiesel properties and also the type of process to be used. It is importance to notice that low-cost raw materials usually contain significant

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amounts of free fatty acids (FFA), which lead to a complex and more expensive final process, e.g. the catalyst depletion is accelerated, the purification costs are increase, and the yield in alkali-catalyzed transesterification is decreased.In the other hand, processing costs could be reduced through simplified operations and eliminating decreased. On the other hand, waste streams. There are several current biodiesel technologies that tried to overcome the issues just indicated. For instance, some plants in Europe produce biodiesel by transesterification using supercritical methanol without any catalyst. In this case, the reaction is very fast (less than 5 min) and the catalyst absence decreases downstream purification costs. However, the reaction requires very high temperature (350–400 ºC) and pressure (100–250 atm) which, in turn, increases the capital and safety costs. Another suggested alternative is the use of heterogeneous catalysts that can be separated more easily from reaction products, and required less harsh reaction conditions than the supercritical methanol process. However, these technologies are still far to produce low cost biodiesel, even if they overcome some problems of the conventional process. In this scenario, new technologies are still required for the transformation of second and third generation biomass raw materials, as well as residual biomass, in sustainable production of biodiesel.

To this respect, recently, an increasing number of applications of ultrasonic processes(US) in chemical transformations have made sonochemistry an attractive area of research and development [13]. The main benefit of US is to enhance chemical reactivity by providing enough energy through out the cavitation phenomenon. The bubble implosions generated in this phenomenon provide sufficient energy to break chemical bonds. Thus, the application of US can completely change the reaction pathways as well as the reaction yield and selectivity. Importantly, the main benefits that can be pointed out from the application of US are the reaction rate increase and the use of less severe operating conditions, as well as shorten induction periods and reduction of reagents amount. An interesting extension of US is the possibility to apply it for the transesterification of vegetable oils to produce biodiesel. Typically, this reaction is kinetically slow and shows mass transfer limitations. Thus, cavitation phenomenon of the US could provide the activation energy required in the reaction as well as the conditions (i.e., mechanical energy) to improve the reaction mixing. In this way, US could provide technical an economic advantage for biodiesel production, as compared to conventional transesterification processes.

In this chapter we report the advantageous application of US for biodiesel lab scale production from *Jatropha curcas* oil (JCO). This proposal is in agreement with the search of optimized, sustainable biodiesel production. The chapter briefly describes the basics of current biodiesel technologies and, in more detail, the fundamentals and benefits provided by sonochemistry to alkaline transesterification process (“sonotransesterification” process). In addition, the experimental setup used for sonotranseterification and the main results to date are also discussed. In general, sonotranseterification shows a significant improve when applied to biodiesel production from JCO; when using a 4.5:1 molar ratio of alcohol/JOC, 25 ºC, atmospheric pressure and 60% of amplitude, yields up to 98% are obtained. Finally, these results are compared to more conventional processes such as supercritical methanol and heterogeneous catalysis for the same raw material. Results are discussed in terms of the ad-

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vantages/disadvantages of reaction operating conditions, energy demand and process time. Notoriously, sonotransesterification shows significant benefits as compare to conventional technologies, which could be further improved as the process be optimized.

2. Biodiesel

Biodiesel is obtained by transesterification reaction, also known as alcoholysis; in this reaction, vegetable oils (preferably non-edible oils) or animal fats are reacted with a significant excess of alcohol (methanol or ethanol), in the presence of a catalyst (homogeneous, heterogeneous or enzymatic), to form fatty acid alkyl esters (FAME) and glycerol, a valuable by-product for industry [2]. In a conventional biodiesel process (CBP), the alcohol-FAME phase is separated and the alcohol excess is recycled (Figure 1). Next, esters undergo a purification process, consisting of water washing, dry vacuum and subsequent filtering. In this process, importantly, the oil used as raw material must be cleaned and its FFA content must belower than 0.5wt%; otherwise, a pretreatment of the raw material must be carried out. Then, the oil is typically mixed with the alcohol in a 6:1 molar ratio, and 1 to 3% homogeneous catalyst (KOH or NaOH) is added to the reaction mixture. Reactants, including the catalyst, must be anhydrous to avoid soap formation. The reaction is then stirred for 40 to 60 minutes, at temperature between 50 and 60 °C, afterward the reaction is completed [2].

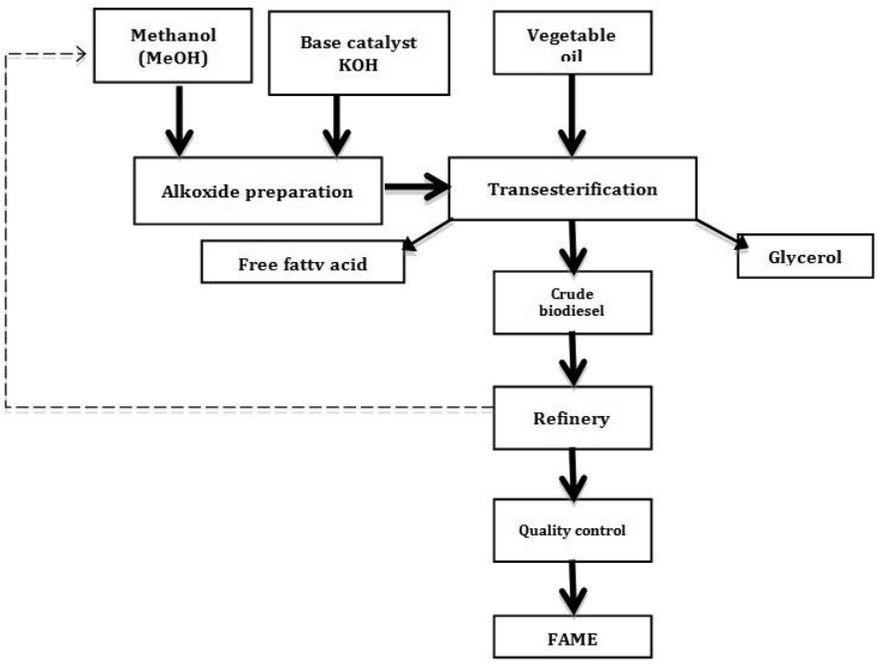


Figure 1. Flow diagram of conventional alkaline homogeneous process for biodiesel production [2].

The overall transesterification chemistry involves an exchange between the alcohol groups (i.e., methanol or ethanol) and glycerol, at given reaction conditions, to produce methyl or ethyl fatty acidsesters (Figure 2). Each fatty acid molecule has the same chemistry configura-

tion [3] and it only differs from other molecules for the carbon chain length or its unsaturation number, which leads to produce FAME with different properties that, in turn, impact the final biodiesel characteristics such as melting point, oxidation stability, etc. This is the reason why the raw material quality is suggested to be the key point for the biodiesel process. Figure 2 also shows the well-accepte dreaction pathway. From the thermodynamic point of view, triglycerides and methanol are well-accepted reaction pathway unable to react at room temperature and atmospheric pressure (i.e., 25ºC and 1 atm, respectively) because of the extremely low solubility of the alcohol into the oil; for this reason, catalysis plays an important role for the alcoholysis reaction to take place.

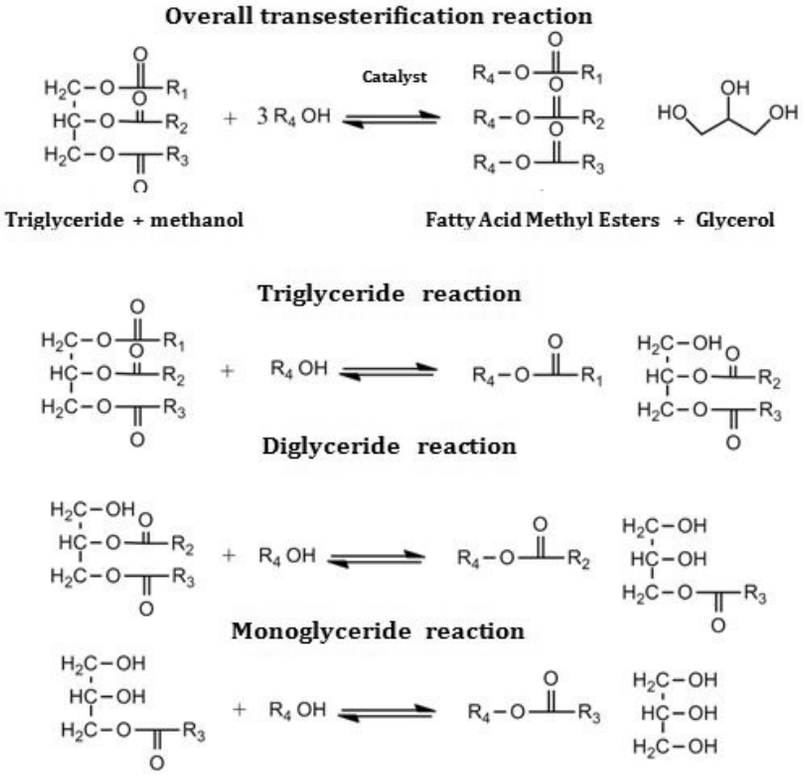


Figure 2. Well-generalized transesterification pathway [3].

Literature [1-4] describes that the first reaction step is the formation of an alkoxide ion (RO^-) through proton transfer from the alcohol. Actually, when homogeneous Brönsted basic catalysts (i.e., NaOH, KOH, Na_2CO_3) are interacts with the alcohol, the following reaction occurs:

$$KOH + CH_3OH \Rightarrow CH_3O^- + [K(OH)H]^+ \quad (1)$$

This alkoxide group then attacks the carbonyl carbon atom of the triglyceride molecule to form a tetrahedral intermediate ion (step 2); therefore, an alkoxide ($NaOCH_3$, $KOCH_3$) is often directly used as catalyst. This intermediate ion rearranges to generate a diglyceride ion

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and alkyl ester molecule (step 3). Next, the diglyceride ion reacts with the protonated base catalyst, which generates a diglyceride molecule and returns the base catalyst to its initial state (step 4). The resulting diglyceride is then ready to react with another alcohol molecule, there by maintaining the catalytic cycle until all the glyceride molecules have been complete converted to biodiesel at 60-80 ºC (Figure 2).

The conventional process (based on homogenous catalysts) has associated several problems, which makes it more expensive when compared to fosil-derived diesel, e.g. raw materials pretreatment and process and cost issues. If raw materials are taken into account, fat and oils cannot directly be used when large amounts of FFA are present. As previously indicated, when alkaline homogeneous catalysts are used, FFA should be less than 0.5 w/w% to avoid high soap formation. Moreover, expensive refinery steps are associated to separate the catalyst and the methanol/biodiesel/glycerol mixture. Generally, water is used to remove alkaline catalysts but this stage makes the overall process less important from environmental point of view. Other relevant issues such as reaction time, mass transfer limitations, optimized set of operating conditions (temperature, pressure, alcohol: oil ratio), determine the economic success of biodiesel production. Regarding the reactor technology, continuous biodiesel process (CBP), especially when equipped with tubular reactors, are always preferred as compared to batch processes. Obviously, this is due to the fact that CBP allow the processing of higher amounts of raw material. However, always that CBP is selected it should be considered the need to incorporate a centrifuge process for glycerol/biodiesel separation, which has a considerable increase in the processing cost. Therefore, an optimum conventional biodiesel process should be conducted at room temperature, atmosphere pressure, avoid water for homogeneous catalysts recovering, and use a low cost glycerol/biodiesel coalescence unit; importantly, the process should reached oil yields over 98%.

Table 1 shows some a comparison of current biodiesel technologies; it is evident from this table that there is a direct connection between complexity and process cost and the quality of final product. As outlined above, conventional process capital cost is low, but processing cost is high because of long reaction time, and separation and purification issues, among others. Regarding the supercritical methanol process, it seems simple and delivers high purity product but, also, capital and operating cost are too high because they are related to severe process conditions. With respect to the use of heterogeneous catalyst, it certainly improves the products separation and purification but, again, this technology is still far to be asuitable economic option because of the high temperature and long reaction time still required for the process. Moreover, another issue to overcome is the design of solid catalysts with appropriate acid sites configuration to improve yield and selectivity, and to decrease catalyst deactivation in hydrous conditions. On the other hand, the US process is less extended and its advantages have not totally documented. However, it could be postulated that the thousands of bubbles formed during the cavitation phenomenon of the US facilitates the formation of a methanol-KOH/oil microemulsion at high temperature, which drastically decreases mass transfer limitations. In this scenario, the transesterification reaction could be carried out within a few seconds, at room temperature (at the "bulk") and atmos-

pheric pressure, thus helping to decrease the process cost. The next section of this chapter describes the basic principles of ultrasound applied to transesterification reaction.

Variable	Homogeneous Catalysis	Heterogeneous Catalysis	Enzymatic Catalysis	Non Catalytic SMP[2]
Reaction time	0.5-4h	0.5-5.5h	1-8h	120-240s
Operation conditions	0.1 MPa, 30-65 ºC	0.1-5 MPa, 30-200 ºC	0.1 Mpa, 35-40 ºC	>25Mpa, >239.4 ºC
Catalyst	Acid/base	Metal oxides o carbonates	Lipase	Non
Free fatty acid	Soap formation	Esters	Esters	Esters
Water	Interfere	No interfere	No interfere	Act as catalyst to the process
Yield	Normal	Low to normal	Low to normal	High
Purification	Difficult	Easy	Easy	Very easy
Downstream	Water	Non	Non	Non
Glycerol purity	Low	Low to normal	Normal	High
Process	Complex	Normal	Simple	Simple
Capital cost	Low	Medium	High	Very high
Operation cost	High	High	Normal	High

Table 1. Comparison of current biodisel technologies for processing biodisel[1] . Source: [9]. [2] SMP: Supercritical methanol process

3. Principle of Ultrasonic Process

Traditionally, sound is a subject studied in physics and it is not a well-met topic in a chemistry course and, so, is somewhat unfamiliar to practicing chemists. However, sonochemistry, which is defined as the use of sound to promote or enhance chemical reactions, has recently received much attention in several chemical reactions concerning sustainability process [5].

It is known that an acoustic wave is a propagation of pressure oscillation in a given medium (gas, liquid or solid), with the velocity of sound producing both the rarefication and compression phases. Figure 3 shows that sound waves are often disclosed as a series of vertical lines or shaded colors, where line separation or color depth represent the intensity or amplitude of the sine wave; the pitch of the sound depends upon the frequency of the wave. According to the sound spectrum, an ultrasonic wave is an acoustic wave whose frequency is above 20 kHz, which is not audible to human. Hence, when a liquid is irradiated by a strong ultrasonic wave, the pressure at some regions in the liquid becomes

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negative (expansion) because the acoustic amplitude of the wave is larger than the ambient pressure. Therefore, if the pressure wave propagating through a liquid has enough intensity, formation of vapor bubbles may occur because the gas dissolved in the liquid can no longer be kept dissolved, because the gas solubility is proportional to the pressure; this is known as the *cavitation phenomenon* [11].

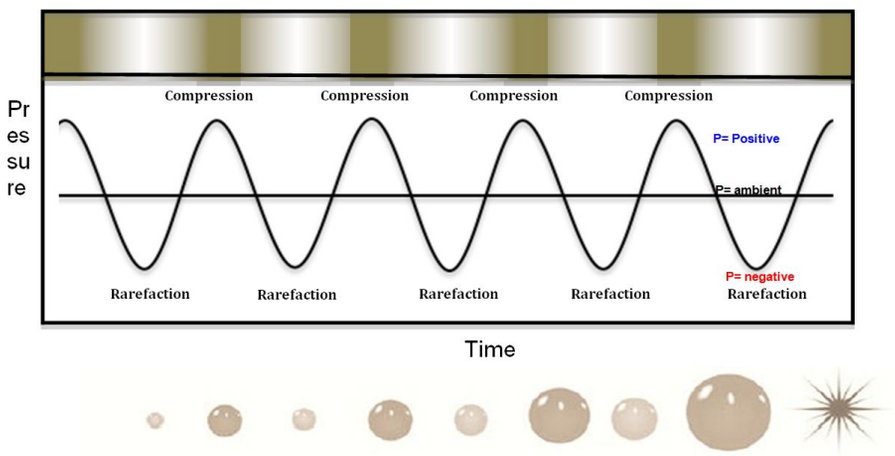


Figure 3. Sound waves interaction with a liquid medium [13]. The bubble growth due to the expansion-compression cycles resulting in the formation of localized "hot spots".

The bubbles formed in the cavitation phenomenon grow from nuclei, over many acoustic cycles, through an elastic process [10]. During the expansion cycle an inflow occurs into the bubble, due to the gradient in gas concentration of the fluid shell surrounding the bubble. As the gas diffusion rate into the bubble is proportional to the concentration gradient of dissolved gas, the net inflow of gas into the bubble is essentially higher during the expansion process. Then, when acoustic bubbles reach a critical size range they undergo a violent collapse. There are three at least theories to explain the chemical effects arising from the collapse of cavitation bubbles:

1. electrical theory,
2. plasma discharge theory and
3. super-critical theory.

Another approach is the "hot spot" theory. This theory suggests that bubbles growth is almost adiabatic up to the collapse. At this point, the gas in the bubble core is rapidly compressed (life time in the order of nanoseconds); hence, temperature of thousands of degrees and pressure of more than hundreds of atmospheres can be locally generated; this is the "hot spot" condition. It is noteworthy that, in addition to the extreme conditions of the "hot spot", a secondary region formed by a thin layer of the liquid surrounding the collapsed bubble, it is also transiently heated, although to a lesser extent; this thin layer is about 200 nm in thickness and may reach a temperature of 1726 ºC [11], see a simplified scheme of the "hot spot" model is shown in Figure 4.

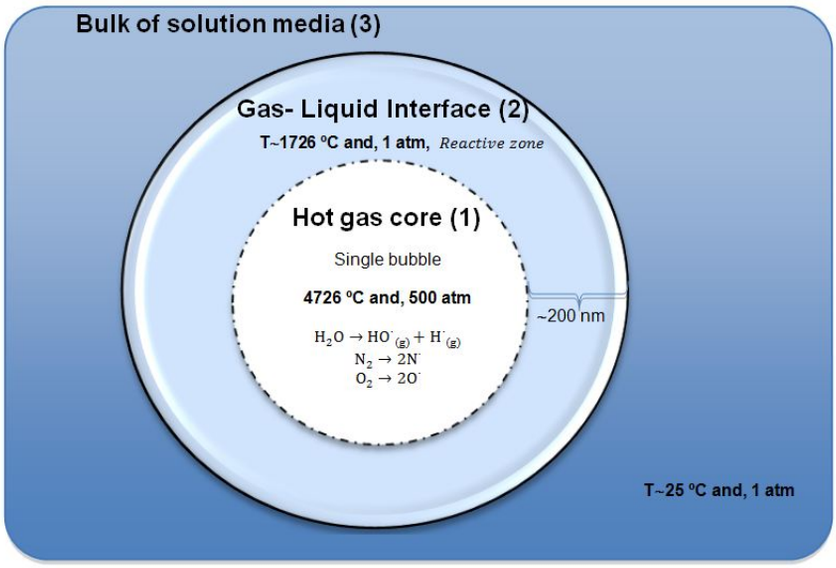


Figure 4. Hot-Spot model in the cavitation process [11].

The physicochemical properties of the solvent and solute, and also the gas in the bubble, have notorious effects on the cavitation phenomenon. Therefore, the sonochemical process is very complicated; it is more frequently influenced by the solvent because cavities are spontaneously formed with solvents having high vapor pressure, low viscosity, and low surface tension. Consequently, as liquid must overcome intermolecular forces to form bubbles, poor cavitation efficiency is obtained when solvents with low vapor pressure, high viscosity, surface tension and density are used. Nevertheless, these kinds of solvents have higher threshold for cavitation but more harsh conditions once cavitation begins; this might help in some chemical reactions [12]. On the other hand, there are several gas phase properties that affect sonochemical cavities, Adewuyi [13] recently reported that heat capacity ratio (also known as polytropic ratio, γ), thermal conductivity and solubility are the most important gas properties. γ is involved with the amount of heat released and, hence, affect the final temperature and pressure produced in the adiabatic compression, according to the following equations [14, 15]:

$$T_{max} = T_0\left[\frac{P_a(\gamma - 1)}{P_v}\right] \tag{2}$$

$$P_{max} = P_v\left[\frac{P_a(\gamma - 1)}{P_v}\right]^{\left[\gamma/\gamma-1\right]} \tag{3}$$

Where T_0= bulk medium temperature, P_v= pressure in the bubble when bubble size is maximum or vapor pressure of the solution, P_a= acoustic pressure in the bubble at the moment of collapse.

Thus, a gas with high thermal conductivity improves the heat transfer from collapsed bubbles to the liquid; this means that it reduces the temperature achieved in an implosion. The solubility of the gas in the liquid is also relevant. The more soluble the gas, the more likely it is to diffuse into the cavitation bubble. Soluble gases should originate the formation of larger number of cavitation nuclei and extensive bubble collapse, because these gases are readily forced back to the liquid phase. Therefore, a decrease of the bulk liquid temperature increases the rate of sonochemical reaction, unlike most chemical reaction systems. This is reasonable because the amount of dissolved gas increases and the vapor pressure of the liquid decreases and, then, less vapor diffuses into the bubble thus cushioning the cavitational collapse; in this condition the implosion more violent.

4. Sonochemical transesterification reaction

There are many aspects that make different the sonochemical and conventional chemical reactions. As already mentioned, the "hot spot" is a suitable concept to explain experimental results in many environmental sonochemistry reactions. This theory considers that reactive species and huge heat are produced from bubble cavitation; each bubble created from the interaction of the ultrasonic wave with the liquid is assumed to be a well-defined microrreactor [13]. Actually, according to the "hot spot" model there are three reactive zones:

1. a huge hot gas core,
2. a gas-liquid interface of approximately 200 nm, and
3. the bulk of the liquid media.

This model is frequently used in aqueous reactions, where solvent or substrate suffer homolytic (symmetrically) bond breakage to produce reactive species, and it assumes that free radicals may be in all the reactive zones. However, this model does not necessarily correspond to the thermodynamic reality of the transesterification reaction, because the system is constituted mainly of triglycerides (TG), small amount of FFA, KOH and methanol. Again, the methanol/oil phase is immiscible creating very large mass diffusional problems. But, in general, the energy generated by the US process produces free radicals, which are very reactive, and a significant amount of heat that improves mass transfer among phases [4]. This combined effect of very reactive species and intimate contact between phases could certainly improve the transesterification reaction rate. In this section, some ideas that further explain experimental results obtained in our laboratories are also discussed.

Figure 5 shows the sono transesterification model, which is an adaptation of Adewuyi´s model [13], constrained as follows.

1. Water hydrolysis is not considered as reactive species source because anhydrous conditions should be achieved for biodiesel processing; from our experience with JCO (FFA <1.5-5%), soap formation is not promoted.

2. Relative humidity of air is also dismissed; so, air is dissolved in the methanol and oil phases.

3. The supercritical theory recently proposed by Hua et al. [16] regarding to the transient supercritical water (373 ºC, 22.1 MPa) at the bubble-solution interface is also discarded, because under these conductions the interphase would be considered as a supercritical methanol microrreactor, and then the use of catalyst would become censer.

However, from our lab experience the sonication of methanol/oil mixture without alkaline catalysts does not produce FAME.

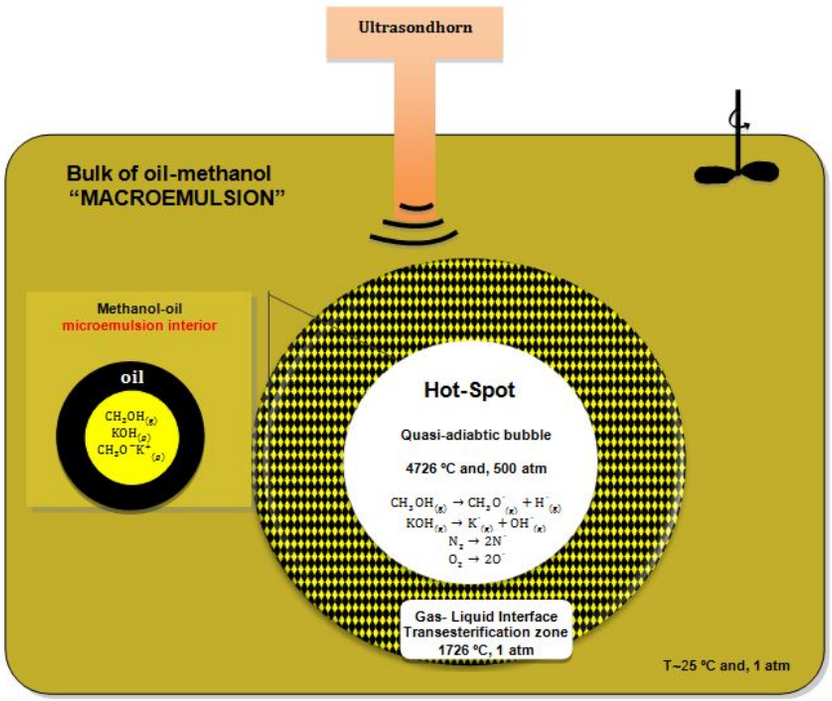


Figure 5. Transesterification cavitation model

The model depicted in Figure 5 assumes that a homogeneous methanol/oil macroemulsion is formed by mechanical mixing. It is very important to note that prior to the sonolysis, the methoxide ion produced, unreacted KOH, and methanol coexist inside the microemulsion. Once that the sound-macroemulsion interaction begins, cavitation is performed with vapor of methanol-KOH and air gas inside the bubbles, carrying out the dissociation reactions of the vapor and gas constituents; then, after several cycles of rarefication and compression, the implosion takes a place involving a significant rate of heat and mass transfer. The surrounding liquid quickly quenches a short-lived, localized entity exposed to high temperature (4226-4726 ºC) and pressure (over 1000 atm). Quenching occurs in few microseconds [17] and very fast cooling rates (about $10^{10º}C^{-1}$). This process has a profound influence on the physical properties of interface (microemulsion), where the transesterification reaction is spontaneously carried out, at local temperatures *ca.* 2000 K, without any diffusional problems.

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As already mentioned, TG transesterification by basic catalysis consists of three consecutive, reversible reactions (Figure 2). In the reaction sequence, TG is converted stepwise to diglyceride, to monoglyceride and, finally, to glycerol, accompanied with the liberation of an ester at each step. The reaction mechanism of TG transesterification shown in Figure 6 indicates that in the catalyst-TG interaction the key step is the nucleophilic attack of the alkoxide ion, originating a different reaction chemical. The conventional transesterification process has been associated to a mass-transfer controlled regime occurring at the beginning of reaction. In addition, as the reaction proceeds and ester products act as emulsifiers, two rate-limiting steps change over time. One step is kinetically controlled and it is characterized by a sudden surge in product formation; the second step is reached once equilibrium is found near the reaction completion [19]. Importantly, in the sono transesterification model showed in Figure 5, neither mass transfer nor kinetic reaction are rate-limiting steps, but rather the chemical equilibrium.

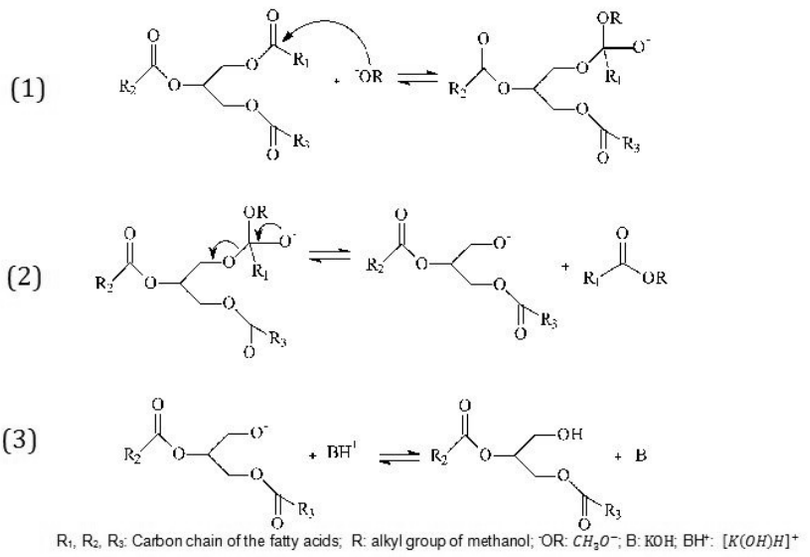


Figure 6. Homogeneous based-catalyzed reaction mechanism for triglyceride (TG) transesterification: methoxide ion form by dissociation of potassium hydroxide into methanol and it is encapsulated into TG-methoxidemicroemulsion, then: (1) CH_3O^-attacks nucleophilically to carbonyl group on TG, which leads to the tetrahedral intermediate formation; (2) intermediate breakdown; (3) regeneration of CH_3O^- active species. These steps are repeated twice to complete TG transesterification, according to Loreto et al. [18].

5. Comparison of experimental biodiesel processing technologies from *Jatropha curcas*

5.1. Why *Jatropha curcas*?

Current feedstock for biodiesel production plants derive from a great biomass variety, including first generation biomass raw materials such as vegetable oils (e.g., soybean, cottonseed,

palm, peanut, rapeseed/canola, sunflower, safflower and coconut oils), animal fats (usually tallow) as well as spent or waste oils (e.g., used frying oils). But, given the fact that the use of vegetable oils has been strongly questioned, the use of second- and third-generation biomass feedstock is continuously growing. Among the raw materials coming from nonedible crops for humans, a key issue is their availability near to the biodiesel production plant. In this scenario, our research group is interested to use *Jatropha curcas* oil as feedstock. *Jatropha curcas L. (JC)* is a stress-tolerant ruderal, drought-resistant, oil-bearing small tree, which is well adapted to tropical, semi-arid regions and marginal sites. *JC* propagates easily and can be established quickly in a wide variety of soils with different agroclimatic conditions and does not put pressure on fertile agricultural land or natural ecosystems. In addition, *JC* is characterized for a short gestation period, low seed cost and, importantly, for the multiple uses that may have different parts of the plant [20, 21]. *JC* has received a lot of attention as a source of renewable energy, because its seeds contain 27–40% nonedible oil with a high quality of fatty acid profile (Table 2), which can be easily converted into biodiesel that meets American and European Standards (Table 3).

Fatty acid	Systematic name	Structure	wt %
Lauric acid	Dodecanoic acid	C12	-
Mysteric acid	Tetradcanoic acid	C14	0-0.1
Palmitic acid	Hexadecanoic acid	C16	14.1- 15.3
Palmitolileic acid	Cis-9-hexadecanoic acid	C16:1	0-1.3
Stearic acid	Octadecanoic acid	C18	3.7-9.8
Oleic acid	Cis-9-Octadecanic acid	C18:1	34.3-45.8
Linoleic acid	Cis-9-cis-12-Octadecanoic acid	C18:2	29-44.2
Linolenic acid	Cis-6-cis-9-cis-12-Octadecanoic acid	C18:3	0-0.3
Arachidice acid	Ecosanoic acid	C20	0-0.3
Behenic acid	Docosanoic acid	C22	0-0.22
Gadoleic acid		C24	14
Saturated	**-**	**-**	**21.1**
Unsaturated	**-**	**-**	**78.9**

Table 2. Fatty acids profile of *Jatropha curcas* oil [22]

In terms of availability, JC easily grows in Norwest Mexico, where our lab is located. For this reason, we set a research project to evaluate the potential of the local JC variety as source of renewable energy (i.e., biodiesel production). Notoriously, we also seek the utilization of valuable byproducts or residues of the conversion of *JC* oil to biodiesel; for instance, fruit husk and seed shell may lead to production of energetic pellets, part of the harvested seed shell may be used to produce humic acid, a biofertilizer; from the seed kernel, not only oil and subsequently biodiesel may be produced, but also protein flour for poultry, sheep,

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shrimp and tilapia (Figure 7). Once that biodiesel is produced a significant amount of glycerol become available, and we look for the production of high-added value chemical derive from glycerol catalytic conversion. Results presented hereby concern to biodiesel production, in particular the development of alternative strategies to improve the efficiency of the transesterification reaction and to decrease the overall processing cost. In this way, the proposed integrated approach clearly contributes for the development of sustainable biomass conversion processes.

Parameter	JC Biodiesel	Diesel	USA ASTM	Europe EN
Density at 15ºC (g mL^{-1})	0.91	0.85	0.88	0.8-0.9
Kinematicviscosityat 40°C (mm^2s^{-1})	3.43	2-8	1.9 - 6	3.5 - 5
Cetane number	52	47.5	"/47	>51
FAME content (%)	>99	0	-	> 96.5
Sulfur (ppm)	0	<5	<15	< 10
Flash point (ºC)	186	>61.5	>93	>101
Acid number (mgKOHg^{-1})	Depend of process		≤ 0.5	≤ 0.5

Table 3. USA and Europe international standards for biodiesel [23].



Figure 7. Productive chain for non-toxic *JC research p*roject in Norwest Mexico [23].

5.2. Materials and methods

5.2.1. Physical chemical JC oil characterization

The study used *JC* from selected elite germoplsms and cultivated in three zones of Sinaloa, Mexico. The approach used to obtain *Jatropha curcas* oil (JCO) was the well-established cold pressing followed by solvent oil extraction. The JCO physicochemical properties studied in this work included: fatty acid profile, acid index (AI), saponification index (SI), peroxide index (PI), and iodine index (II), which were obtained following the methodologies suggested by the Association of Analytical Communities, AOAC.

The quality criteria for the production of biodiesel are specified in EN 14214. In particular, method EN 14103 specifies the FAME content, which is used to profile the vegetable or animal oil feedstock used in biodiesel production. EN 14103 requires calibration of all FAME components by relative response to a single compound, methyl heptadecanoate. This requires the measurement of accurate weights for each sample and the addition of an internal standard. The FAME range for which the method is intended lies between C14:0 and C24:1. A modified EN 14103 chromatographic method was used. In this method, FAME analysis was carried out in a 6890N Agilent Gas Chromatograph (GC), equipped with a capillary split/splitless injector and a selective 5973 Agilent mass spectrometer detector. A 1 μL split injection (split ratio 50:1) was made to a Supelco omega wax column (bonded polyethylene glycol), using 1 $mlmin^{-1}$of helium into the column as carrier. Samples were injected via an auto sampler series 7683 also from Agilent technologies. A good resolution and peak shape was obtained when using the following oven temperature program: The initial temperature, 100 °C was kept for 2 min; then a heating rate of 4 °C min^{-1} was used to increase the temperature to 240 °C and, finally, this temperature was kept for 10 min. For identification and calibration of the individual FAME, the Supelco standard "37 Component FAME Mix" was used. The response and retention time of each component was experimentally determined. Then, the calibration was verified by both, the analysis of a calibration-check standard and the database of mass spectrum reported by the National Institute of Standards and Technology (NIST). Results of analyses were then compared with the certificate of analysis, verifying the quality of the calibration. The standard preparation for this technique consisted of the dilution of the FAME standard into 4 mL of n-heptane. The sample preparation was also quite simple with 100 μL of biodiesel feedstock into 4 mL of n-heptane. Finally, concentration reports were based on the area percentage rather than a mass percentage, to simplify the calculations.

On the other hand, quantitative determination of free and total glycerin in biodiesel (B100) was also carried out by gas chromatography, followed by a modified methodology proposed by the ASTM D6584-10aε[1].The same Agilent GC system was also used for this analysis, the only difference being the use of a MS detector. ADB-5 ms column from Agilent Technologies was used for free and total glycerin analysis, which is equivalent in chromatographic efficiency and selectivity to that of the MET-Biodiesel capillary column of Sigma Aldrich.

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5.2.2. Transesterification procedure

5.2.2.1. Conventional process

Conventional alkaline transesterification was conducted in a 2-necked glass reactor (100 mL, Aldrich). A homogeneous reaction mixture was obtained by using plate stirrers, and a constant reaction temperature was kept by using isolated bath vessels equipped with a stainless steel coils. The reaction temperature was fixed by using of a heater/cooler recirculation isothermal bath (Fisher Scientific 3016). Figure 8 shows that each reactor was connected to-cooled straight glass condenser to avoid alcohol leaks; water at 5ºC from another isothermal bath (Fisher Scientific 3028) was used as cooling fluid.



Figure 8. Transesterification reaction system for the conventional process

Anhydrous methanol (Sigma-Aldrich, 99.8 %,) and KOH reagent grade (Sigma-Aldrich, 90%) were used for all experiment of this study. The stirrer was fixed at 600 rpm, and the temperature at 40, 60, 70 or 90 C), a methanol: JCO molar ratio was 3:1 or 6:1. Previously to each reaction, methanol and KOH solutions were prepared according to the proposed molar ratio. Then, the reaction volume was fixed to 50 mL of JCO. After the desired reaction temperature was reached, a preheated methanol-catalyst solution was added to start the reaction. Reaction mixture was sampled after 15, 45, 60, 90, and 120 min. These samples were quenched by a sudden immersion of the sample to a plastic container at 0 C, for 15 min. Then, reaction products were purified according to the methodology suggested by Cervantes [24](Figure 9), and the biodiesel yield was determined by means of the following equation:

$$Yield = \frac{wt\ B_{100}}{wt\ oil} x100 \tag{4}$$

5.2.2.2. Heterogeneous process

As reported elsewhere [25], JCO transesterification was also conducted by using ZnO, Al_2O_3 and ZnO-Al_2O_3 mixed oxide powders as catalysts. The objective was to compare the heterogeneous catalytic conversion of the same JCO. In this case, the catalytic activity was measured in a Parr 4560 stirred tank reactor, operated at 1000 rpm, 250ºC, P= 14.7 atm. A methanol: JCO molar ratio of 6:1 and a 3 wt% of catalysts (based on JCO weight) were used. Previously to the reaction, the reactor was uploaded with the 50 ml of JCO, the required methanol to achieve the 6:1 molar ratio, and 1.36 g of catalyst. Then, the reactor was purged with nitrogen (Praxair, reagent grade) for 3 min to avoid JCO burning. The reaction time was 1 h and then the reactor mixture was suddenly cooled to room temperature. The product separation included the following steps.

1. Catalysts removal by means of vacuum filtration.
2. Methanol recovery by using a rotary evaporator at the same condition indicated in Figure 9.
3. Glycerol and biodiesel separation by centrifugation, using the same condition indicated in Figure 9.

At the end, the biodiesel yield was calculated by using equation 4

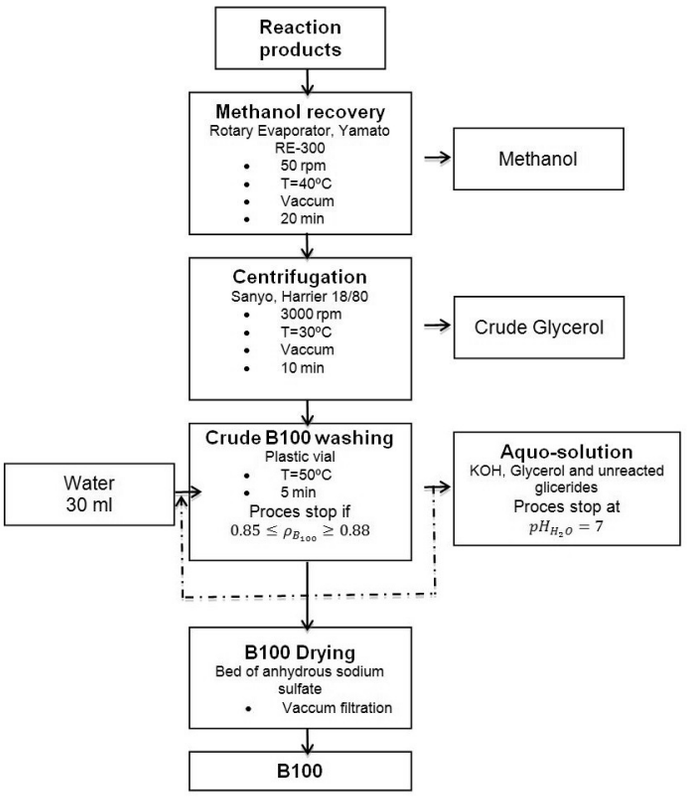


Figure 9. Biodiesel purification process for a conventional alkaline transesterification process.

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5.2.2.3. Supercritical methanol process

Non-catalytic transesterification process was evaluated by means of the supercritical methanol reaction. This process was also carried out using the 4560 Parr stirred tank reactor. The effect of both, methanol: JOC molar ratio (40:1 and 60:1) and temperature (250, 300, and 350ºC) was evaluated using nitrogen as co-solvent. Once the required reagents amounts were charged to the reactor, the air was vented with nitrogen and the stirrer was fixed at 1000 rpm. Next, the temperature was increased until the desired set point; in this process the pressure increased but not enough to reach the methanol supercritical point. Therefore, additional nitrogen was loaded to ensure 14 MPa. As an alternative to decrease the drastic operation conditions $N_2$was used as co-solvent. The reaction took place over 30 min, sampling the mixture every 5 min through the liquid reactor valve. After the reaction was finished, the reactor was suddenly cooled to room temperature. The product separation included the following steps.

1. Methanol recovery by using a rotary evaporator at the same condition indicated in Figure 9.

2. Glycerol and biodiesel separation by decantation.

At the end, the biodiesel yield was calculated by using equation 4.

5.2.2.4. Ultrasonic process

The sonotransesterification of JCO was conducted by using a highly efficient Hielscher Ultrasonic processor, model UP200 HS. This equipment was used to generate mechanical vibrations by means of the reversed piezoelectric effect (electric excitation), with frequency of 24 kHz, and a control range of 1 kHz. The vibrations were amplified by the S14 sonotrode fitted to the horn and formed as a $\lambda/2$ vibrators, and transferred via its end face to the JCO. To optimize the sonotransesterification reaction, the effect acoustic power density (N), sonication time (or reaction time), and methanol: JCOmolar ratio (MR) were evaluated at room temperature (25ºC) and ambient pressure (1 atm). Reaction temperature was controlled by using an isothermal bath (Fisher Scientific 3016). The continuous sonication of the reaction mixture was conducted using N=105 Wcm^{-2} and a molar ratio of 6:1, following the approach described in Figure 9. Reaction time was fixed at 1, 2, 4, 6, 8, 10, 15, 20, 25 or 30 min. Next, the methanol: JCO molar ratio was evaluated varied to 3:1, 4:1 and 6:1. For the smaller reaction time and molar ratio, the acoustic power density effect was evaluated at 42, 63, 73.5, 84, 94.5, and 105 Wcm^{-2}. When the best set of parameters was found, an experiment was conducted again to determine the biodiesel quality.

5.3. Results and discussions

5.3.1. Physical chemical JC oil characterization

The *Jatropha curcas* oil obtained from non-toxic, harvested seed in Northwest Mexico, seems to be an excellent candidate for biodiesel production due to its high quality. Table 4 includes the basic JCO physicochemical characteristics that back up this quality. The iodine index is a measurement of the oils unsaturation degree; a higher iodine index corresponds to higher degree of unsaturation [26], and probably leads to oxidation and viscosity problems. The JCO iodine index was 28.75 cg I_2g^{-1}, which is well below the maximum specified value (120 cg I_2 g^{-1}) for biodiesel as indicated in the EN14214 specification. The limitation of unsaturated fatty acids is convenient because heating higher unsaturated fatty acids results in polymerization of glycerides, leading to the formation of deposits or to deterioration of the lubricant [27]. Fuels with this characteristic (e.g Sunflower, soybean and safflower oil) are also likely candidates to produce thick sludge's in the sump of the engine, when fuel seeps down the sides of the cylinder into crankcase [26]. The JCO iodine index could was caused by the high content of unsaturation fatty acid such as oleic and linoleic acid (Table 5).

Test	Parameter[1]
Appearance	Yellowish transparent
Free fatty acid (%)	1.51 ± 0.10
Density at 15ºC (gml^{-1})	0.92 ± 0.01
Acid index (mg KOH g^{-1})	3.07 ± 0.12
Saponification index (mg KOH g^{-1})	180.92 ± 2
Iodine index (cg I_2 g^{-1})	28.75 ± 0.1
Peroxide index (meq O_2 Kg^{-1})	18.5 ± 0.7

Table 4. Physical chemical properties of *Jatropha curcas* Oil. [1] Standard desviation measured from triplicate determinations.

In on another hand, JCO peroxide index was 18.5 $meqg^{-1}$, that is higher than the index recently reported in the literature for crude seed Jatropha oil, 1.93 $meqg^{-1}$ [26] and 2.5 $meqg^{-1}$[28]. Despite this high peroxides index, JCO upholds the good quality of biodiesel purposes. The JCO saponification index was 181 mg KOH g^{-1}, which suggested that JCO was mostly normal triglycerides, and very useful in biodiesel production due to its low FFA content (1.15wt%). The content of FFA was assessed from the acid index (AI) measurement, taking into account the composition showed in Table 5. The acid index of 3.07 mg KOH g^{-1}reported in Table 3 was lower than the values reported by other authors (10 - 14 mg KOH g^{-1}) for crude JCO [29, 30]; this could be attributed to the change of local environmental conditions where by the *Jatropha curcas* plant was grown. Therefore, acid index becomes a very important parameter to determine the most convenient

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processing route of a given FAME; this means that oils can undergo a pretreatment or direct transesterification as a function of FFA amount.

Compound	Estructure	wt %
Palmitic	16:00	23.992
Stearic	18:00	7.224
Oleic	18:01	41.368
Linoleic	18:02	27.186

Table 5. Faty acid composition de *Jatropha curcas* Oil determine by MS-CG.

The properties of triglyceride and biodiesel are determined by the amounts of each fatty acid present in the molecules. Chain length and number of double bonds determine the physical characteristics of both fatty acids and triglycerides [3]. Nevertheless, transesterification does not alter the fatty acid composition of the feedstocks, and this composition plays an important role in some critical parameters of the biodiesel, as cetane number and cold flow properties. Therefore, measuring fatty acid profile of JCO was another important target of this study. These results are shown in Table 5.

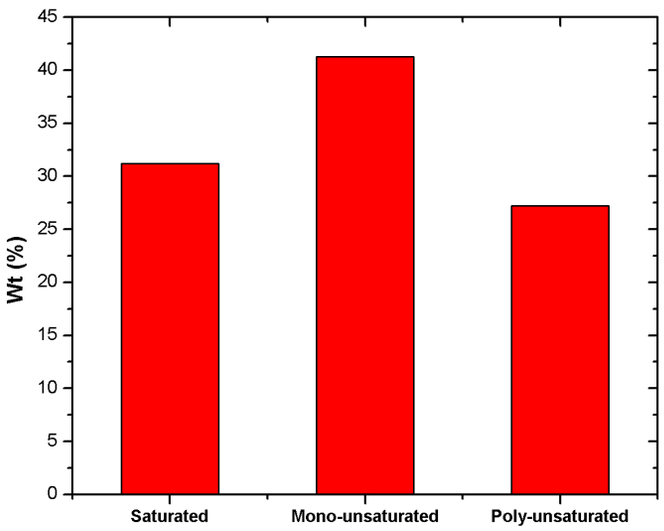


Figure 10. Type of fatty acids in *Jatropha curcas* oil from the Norwest of México

There are three main types of fatty acids that can be present in a triglyceride which is saturated (Cn:0), monounsaturated (Cn:1) and polyunsaturated with two or three double bonds (Cn:2,3). Ideally, the vegetable oil should have low saturation and low polyunsaturation,

that is, be high in monounsaturated fatty acids, as shown in Figure 10. Vegetable oils rich in polyunsaturated (linoleic and linolenic) acids, such as soybean and sunflower oils [26], usually produce methyl ester fuels with poor oxidation stability. In the other hand, vegetable oils with high degree of unsaturation (Cn:2,3) lead to a product with high freezing point, poor flow characteristics and may become solid (e.g palm oil) at low temperatures, although they may perform satisfactorily in hot climates. The main fatty acids in the JCO used in this study were the oleic, linoleic, palmitic and the stearic fatty acids. The predominant acids were monounsaturated (41.36%), polyunsaturated (27.18%) and saturated fatty acid (31.21%) (Figure 10). This result was in agreement with the reported by Akbar [26], although it was slightly different in terms of saturated and polyunsaturated compounds for the JCO from Malaysia. Thus, JCO can be classified as oleic–linoleic oil. Compared to others vegetable oil JCO had highest oleic acid contain than palm oil, palm kernel, sunflower, coconut, and soybean oil.

5.3.1. Jatropha curcas oil transesterification

Three current biodiesel technologies were evaluated and compared with the conventional homogeneous transesterification, using the JCO characterized above. The main objective was to evaluate the potential advantages of sonotransesterification in terms of operating conditions, transesterification rate and processing steps and costs.

Conventional alkaline transesterification

According to the overall transesterification pathway shown in Figure 1, stoichiometrically, JCO methanolysis requires three moles of methanol for each mole of oil. Since the transesterification of triglycerides is a reversible reaction, excess methanol shifts the equilibrium towards the direction of ester formation. As it is evident from Figure 11, the maximum yield for the conventional alkaline transesterification process (84%) was reached after 15 min reaction time; afterwards no signification variations were observed. In addition, when the methanol: JCO molar ratio was increased from 3:1 to 6:1, no major differences were found within the first 15 min; however, a higher biodiesel yield was observed in the experiment with a 6:1 molar ratio toward the end of the reaction. On the other hand, results shown in table 5 indicate that temperature effect is not important. These results correspond to the biodiesel yield evaluated after 15 min. Thus, the higher biodiesel yield was found at 40 °C, and then it decreased to around 73 – 75 % for temperatures between 60 and 90 °C.

Current results of the conventional process disclosed in Figure 11 and Table 6 suggested a significant improve to the conventional alkaline transesterification process, because the reaction yield was enhanced at a shorter reaction time (40 min as compared 60 min) and temperature (40 °C as compared to 60ºC) for industrial application [3]. A shorter reaction time can be translated to a continuous process with a shorter resident time and then, the possibility to reduce costs at the reaction stage. However, a higher JCO conversion is needed to ensure a sustainable process. Moreover, the biodiesel purification process is still a problem because it implies long times and it is energy demanding.

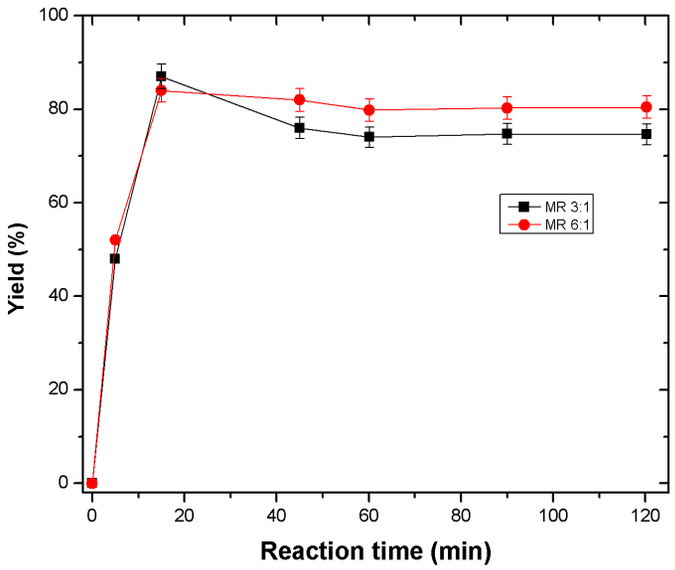


Figure 11. Progress of transterification reaction as function of methanol:JCO ratio at 40ºC

Temperature, ºC	Yield, % (At 15 min of reaction time)
40	84.0
60	73.06
70	73.60
90	75.30

Table 6. The effect of temperature on the performance of alkaline transesterification of JCO by conventional process.

Supercritical methanol process

Thus, as an alternative of the problems indicated above, the supercritical methanol process (SMP), using nitrogen as co-solvent, was conducted. Figure 12 shows that the best set of operating conditions for this non-catalytic process were: methanol: JCO mol ratio of 40:1 and 350ºC. Under these conditions, a biodiesel yield *ca*. 60% was obtained. From Figure 13, it can be observed that after 20 min the equilibrium was reached for the transesterification reaction for both molar ratios studied: 40:1and 60:1. This is a very promising result if it is compared with reported for palm [31] and soybean oils [32], where biodiesel yields up to 84% were obtained under very high pressure, 40 and 35Mpa, respectively.

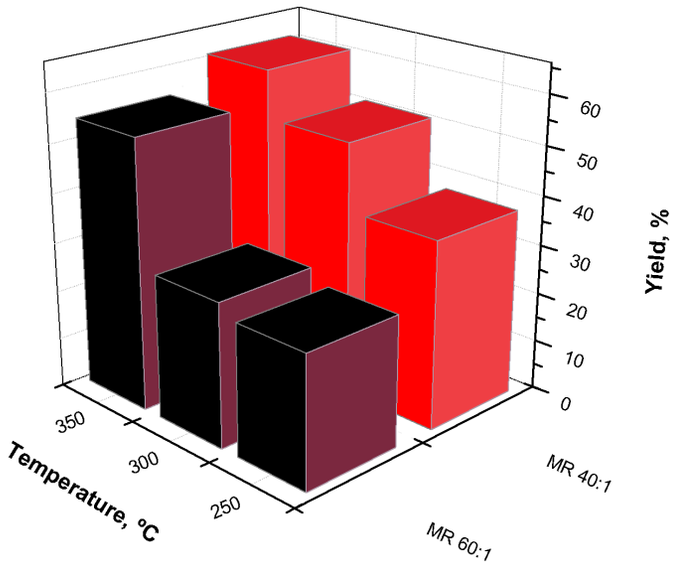


Figure 12. Effect of temperature and methanol: JCO molar ratio on the yield of Biodiesel obtained by supercritical methanol process at 14Mpa and 30 min.

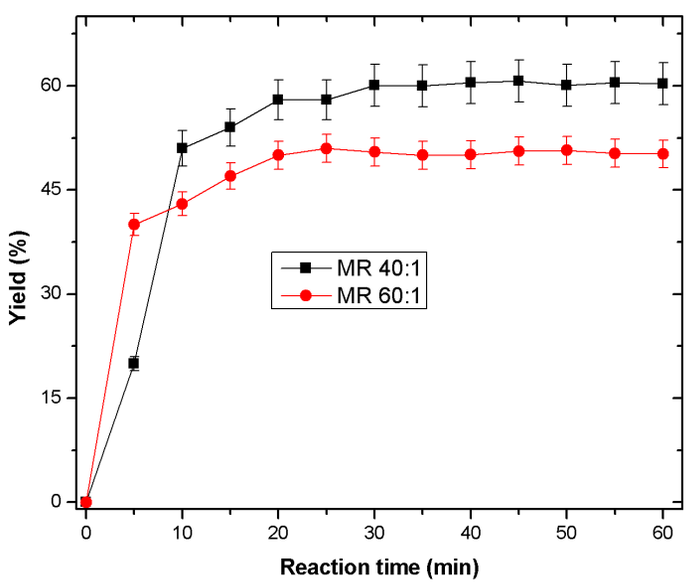


Figure 13. Progress of transterification reaction as function of methanol: JCO ratio at T= 350ºC for supercritical methanol process

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Importantly, supercritical methanolysis did not require any kind of catalyst, and no pretreatment to remove water or FFA was used in this work. A very simple separation processes – evaporation and layer separation – were used for biodiesel purification. Our findings agree with the literature that supercritical process is simpler and faster than conventional alkaline transesterification for biodiesel production. In addition, since wastewater was not introduced by pretreatment or washing processes, the supercritical process is environmental friendly. However, to date, high investment and energy cost are still required due to high temperature and pressure of the supercritical state. Another issue with economic implications is the large methanol needed to enhance the forward reaction without catalyst. It could expected that these costs are comparable to those of the pretreatment and separation process of the conventional alkaline transesterification process. Clearly, as the methanol demand be decreased, and the operating conditions be more moderate, the economic feasibility of supercritical methanol process would be possible.

Heterogeneous process

As indicated in the previous section, three heterogeneous powder catalysts, ZnO, Al_2O_3 and ZnO- Al_2O_3 mixed oxides supported on SBA-15 were evaluated for transesterification reaction. Figure 14 shows our best results to date, when experiments were conducted with a methanol: JCO molar ratio of 6:1, 250ºC, and 3 wt % of catalyst. Results were collected after 1 h of reaction time. Under these conditions, the equilibrium biodiesel yield (83%) was reached for the supported Al_2O_3 catalysts. Importantly, no catalysts deactivation was observed for at least 10 runs (without regeneration treatment). It is noteworthy that $Al_2O_3$is traditionally used as support instead of active phase due to its poor catalytic activity for transesterification [33]. In fact, in our experiments Al_2O_3 itself showed no more than 5% of FAME yield, but the it showed a totally different catalytic performance when it was well dispersed on SBA-15. On the other hand, several supported basic catalysts have also been reported in the literature -sodium [33] or potassium [34] loaded on a support (normally alumina), using several precursors and treated at high calcination temperatures (500–600ºC). The catalysts showed good activities (80-90 % biodiesel yield) at low temperatures (70-90ºC), but no data were reported about their stability. K_2CO_3 supported on both MgO and Al_2O_3 provided good results for rapeseed oil transesterification with methanol at 60–63 ºC, but K_2CO_3 leached into the solution.

Meantime, pure ZnO and ZnO supported on Al_2O_3 have also been reported as good transesterification catalyst. In experiments performed in a packed-bed reactor at 225-230 ºC, 91.4% and 94.3% of FAME yields were obtained, for 1 and 7 h, respectively [35]; in this case, no Zn leaching was practically observed (5 ppm). In addition, no data about catalysts has been reported. In our case, experiments conducted with ZnO and ZnO/Al_2O_3 showed biodiesel yields below 75 %. The most promising results found for the Al_2O_3/SBA15 have to be studied in detail to optimize the catalyst formulation.

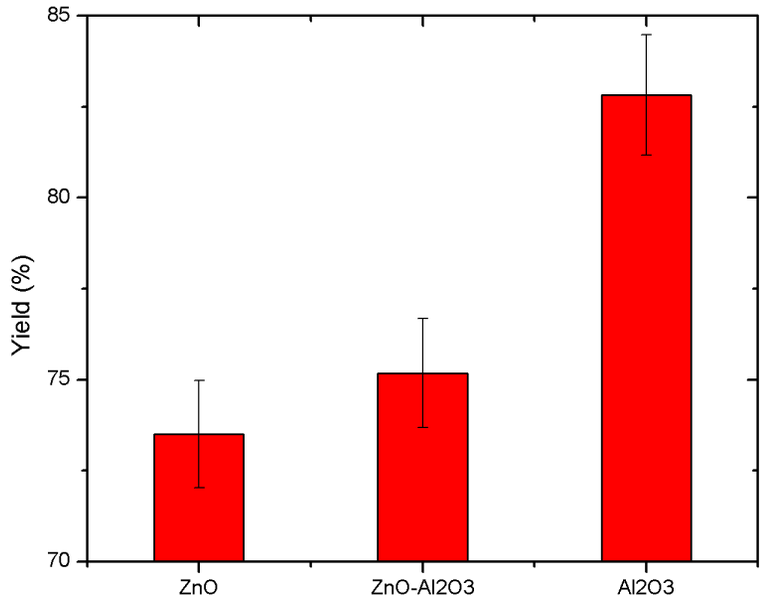


Figure 14. Yield of biodiesel of transesterification of JCO with MR=6, and 250ºC, 1 h and 3 wt.% of each heterogeneous catalyst.

Sonotransesterification

Experimental results of JCO sonotransesterification are shown in Figure 15. The first issue that became evident was that sonotransesterification was much faster than the conventional alkaline transesterification. Thus, in just 1 minute of reaction time the maximum FAME yield (*ca*. 65%) was reached for the experiment conducted with a methanol: JCO molar ratio of 6:1, an acoustic power density (N) of 105 Wcm^{-2} and temperature of 25ºC. Moreover, Figure 16 shows that for 1 min of reaction time, a reduction of the methanol: JCO molar ratio from 6:1 to 4:1 increased the biodiesel yield. Under these conditions, a 71 % biodiesel yield was obtained. Notoriously, the later molar ratio is closer to the stoichiometric one, thus helping to decrease the excess of alcohol required by the other biodiesel technologies under comparison in this study. These results clearly showed the following advantages for the sonotransesterification process: a shorter processing time is required, a lower amount of alcohol is required (almost the stoichiometric amount), and the experiment is conducted at room temperature and atmospheric pressure.

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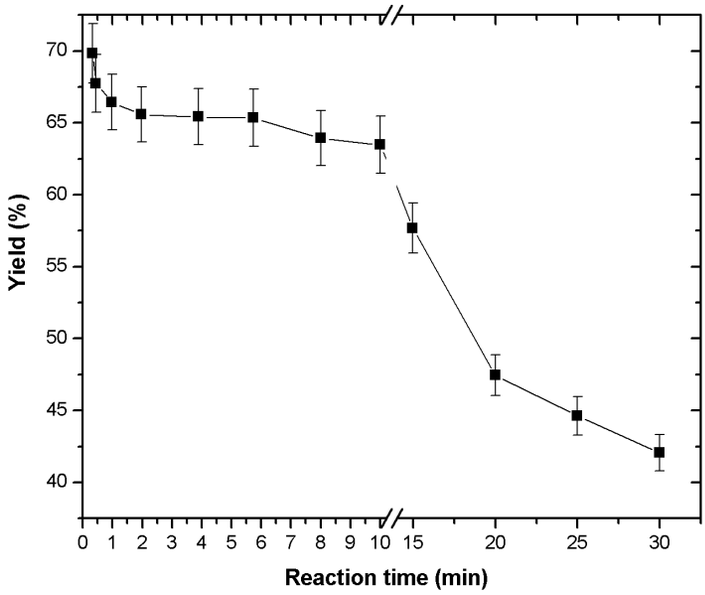


Figure 15. Effect of the sonication time on the yield of biodiesel by sonotransterification reaction with MR of 6:1, room temperature, and acoustic power density of 105 Wcm^{-2}.

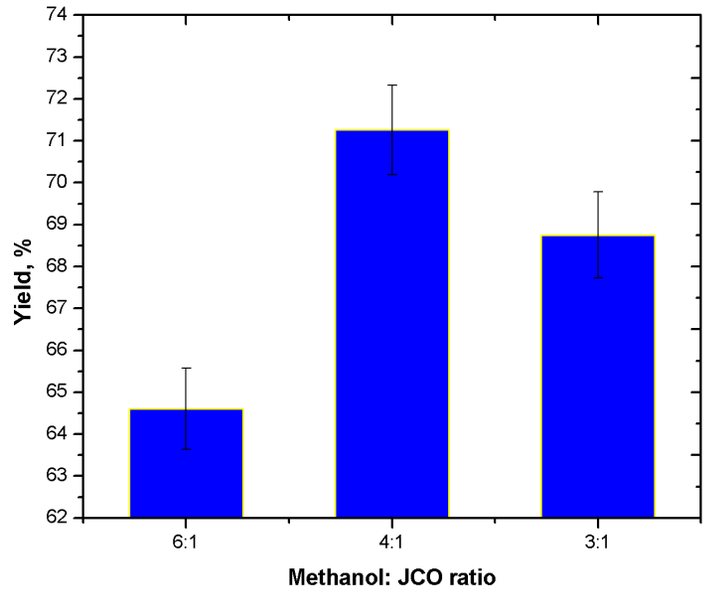


Figure 16. Effect of the methanol:JCO molar ratio on the yield of biodiesel by sonotransterification reaction at room temperature,1 min of reaction time, and acoustic power density of 105 Wcm^{-2}.

Despite of the important advantages initially found for the sono transesterification process in this work, the biodiesel yield had to be increased to make it atractive from the industrial point of view. To this respect, a more detail study of the acoustic power effect was conducted. Figure 17 shows that acoustic sonocation power had a significant effect on yield. For an N of 64 Wcm^{-2}, coupled with the best set of parameters used in previous experiments, a FAME yield up to 96% was reached at room temperature. The reason why a higher transesterification rate was obtained with the ultrasonic process was already outlined in the previous sections. Briefly, the huge local temperature generated in the "hot spot" formed during the cavitation phenomenon favors the formation of highly reactive species and promotes mass transfer. These issues are the key to improve the transesterification reaction rate because under the experimental ultrasonic conditions the process is not affected by mass transfer or by kinetic limitations, but rather by the equilibrium condition.

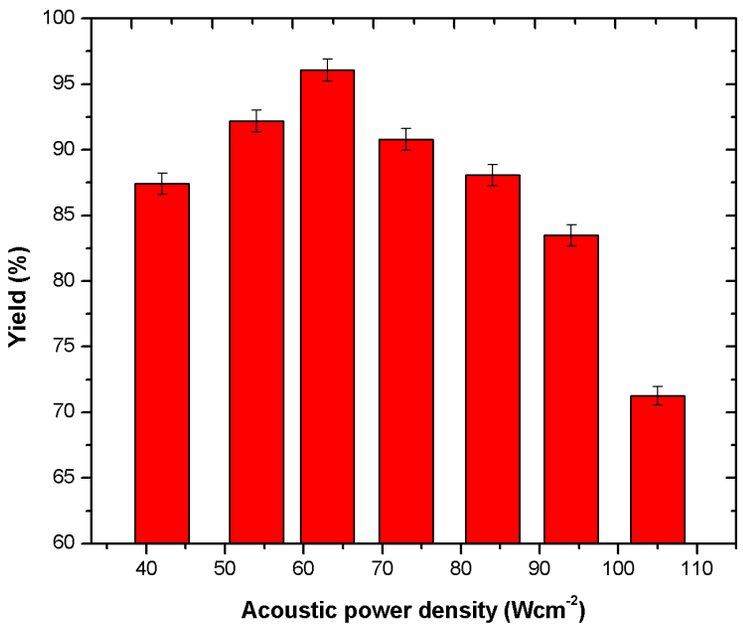


Figure 17. Effect of the acoustic power density on the yield of biodiesel by sonotransterification reaction with MR of 4:1, room temperature, and one minute of reaction time.

In this scenario, sono transesterification becomes a very attractive process to be implemented in a continuous industrial process. Thus, results found in Figure 17 were used to configure a continuous US process with a tubular sonorreactor, using a resident time of 1 min. In this case, a constant yield of 96% was reached. Importantly, the quality of the biodiesel obtained in this experiment, overcame the quality of biodiesel with international standards (Table 7).

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Parameter	Value
FAME content[a] (%)	98
Density at 15ºC (gml^{-1})	0.84
Acid index ($mgKOHg^{-1}$)	0.5
Total glycerin (wt. %)	<0.2
Free glycerin (wt. %)	<0.02

Table 7. Physical chemical properties of biodiesel obtained by sonotransesterification under continuous process with optimized conditions operated at room temperature. [a] after purification process

6. Conclusions

Nowadays, the conversion of non-edible and residual biomass feedstock into biofuels is already considered a suitable alternative for the generation of alternative energy sources. In particular, transesterification of oils and fats is a well-known technology, and the production of biodiesel is continuously growing using second- and third-generation biomass raw materials. The main technology used in the industrial production of biodiesel is based on the alkaline transesterification of vegetable oils with methanol. However, the problems related with this technology (mainly in operating conditions and product purification) are the driving force for research in the field of heterogeneous catalysis for biodiesel production and for the development of non-catalytic process under supercritical fluids. The use of heterogeneous catalysts and supercritical methanol process for transesterification reaction seem to be attractive for industrial application because theses simpler processes have a beneficial impact in the process economy. In particular, industry is making great research efforts to find the optimum catalyst formulation and to decrease the drastic operating conditions of supercritical methanol process. However, these technologies are still far to be economically attractive. A more recent alternative is the new ultrasound-assisted method for biodiesel production, which has to be tested and optimization for this particular application.

In this work, we evaluated and compared the performance of four technologies for the transesterification of JCO obtained from JC grown in Northwest Mexico: conventional alkaline catalyst (KOH), heterogeneous powder catalysts (ZnO, ZnO/Al_2O_3 and Al_2O_3/SBA 15), supercritical methanol and sonotransesterification. Results showed that the ultrasonic method has significant advantages as compared to the other three methods. Notoriously, ultrasonication reduced the transesterification reaction time to 1 min at room temperature and atmospheric pressure, as compared to 1-6 h in conventional processing under more drastic operating conditions. We suggest that this result could be explained with the proposed sonotransesterification cavitational model where by diffusional problems are eliminated. Our results demonstrated that acoustic power density and methanol: JOCmolratio are the most sensitive parameters to increase FAME yield for JCO; at the best set of experimental conditions, the biodiesel yield is higher than that obtained by conventional methods. Importantly, the ultrasound-assisted method was also effectively used for continuous production of bio-

diesel by using a plug flow reactor; the physicochemical properties of the biodiesel produced, such as acid value, density, FAME content, total and free glycerin were within the limits of ASTM and EN standards.

In summary, sonotransesterification is faster and easier to handle than conventional transesterification processes. The sonoreactor is significantly cheaper, and the process works under safer and less energy demanding conditions (e.g room temperature and ambient pressure). The major advantages of the current ultrasonic system include operational simplicity, short reaction time, high conversion and reusability. In summary, ultrasonic irradiation is a faster alternative that leads to higher product yield, and with the real possibility to benefit the process economy. Thus, the ultrasonic process discussed in this work established the basis for the development a sustainable process for biodiesel production, although some issues are still to be solved; for instance, if water is avoided in the purification process,the overall process would be even more environmentally friendly.

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