# Biodiesel Production from *Jatropha curcas* Oil Using Potassium Carbonate as an Unsupported Catalyst

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## Abstract

*Jatropha curcas* (JTC) oil, an inedible vegetable oil, can be a substitute feedstock for traditional food crops in the production of environment friendly and renewable fuel (biodiesel). In the present study, unsupported potassium carbonate was used as a catalyst to provide an understanding of the catalytic activity in the transesterification reaction. Researching the potential and the behavior of potassium carbonate is very important because every biomass ash contains this compound in a significant amount. It can be extracted by using classical extraction or leaching technologies. During the biodiesel production reaction, the formation of soap as a byproduct was also monitored using the FTIR-ATR method. From this study it was observed that the transesterification of JTC oil to JTC biodiesel appeared to be complete within 15 minutes when a 5 wt% (based on the wt. of the oil) potassium carbonate, 6:1 methanol to oil molar ratio, 60°C or a 4 wt% potassium carbonate, 9:1 methanol to oil molar ratio and 60°C reaction temperature were used.

**KEYWORDS:** *Jatropha curcas* oil, transesterification, biodiesel, FTIR-ATR

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# 1. Introduction

The need to meet the increasing global demand for energy while addressing climate change concerns has provided an impetus for research into the production of biofuels. Biodiesel is one of the most attractive biofuels because of its biodegradability, higher flash point, reduced production of most regulated exhaust emissions, miscibility in all ratios with petrodiesel, compatibility with the existing fuel distribution infrastructure and inherent lubricity (Knothe, 2008). Biodiesel is produced by the transesterification of triglycerides (for example, vegetable oils and animal fats) reacted with monohydric alcohols in the presence or absence of base or acid catalysts or enzymes. In this reaction, the fatty acids of the triglycerides are converted to the corresponding ester, which is called Fatty Acid Alkyl Ester (FAAE). This fatty acid alkyl ester is commonly known as biodiesel. When methanol is used as the monohydric alcohol, then the resulting fatty acid methyl ester (FAME) is the biodiesel, as illustrated in Figure 1.
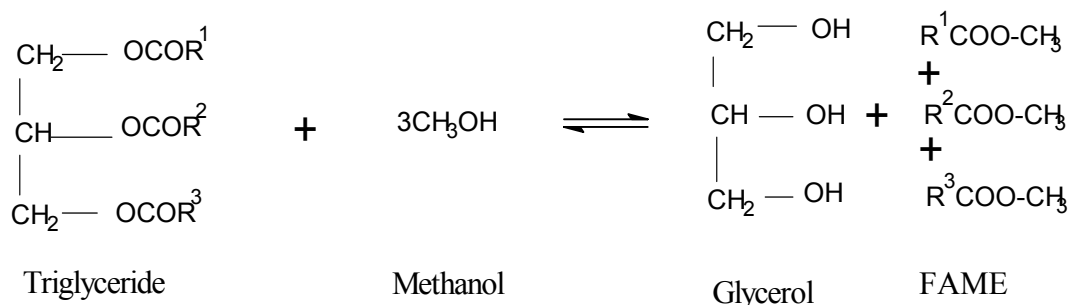


Figure 1. Transesterification reaction where, $R^1$, $R^2$ and $R^3$ represent the different fatty acid derivatives (long unbranched aliphatic tail (chain), which are either saturated or unsaturated)

At present, the United States and Europe are world leaders in biodiesel production. The United States depends mainly on soybean for biodiesel production, while Europe depends on rapeseed. Other traditional feedstocks are canola oil, sunflower oil, palm oil and coconut oil, all of which are food sources. It is now believed that the world food crisis is the result of the utilization of food crops in producing biofuels (Kamal et al., 2008). Human rights activists have called for a ban on the production of biodiesel from food crops for several years. Waste cooking oil and tallow from animal fats can be alternative feedstocks for biodiesel production, but large scale biodiesel production from these sources may not be possible because of the lack of continuous and sufficient supply of these types of feedstock. So, there is the need for an alternative biodiesel feedstock that is inexpensive, inedible and meets all the criteria for biodiesel feedstock.

In this regard, *Jatropha curcas* oil, an inedible tropical plant oil has tremendous potential for biodiesel production. Jatropha oil has low acidity and good oxidation stability compared to soybean oil, low viscosity compared to castor oil and better cold properties than palm oil (Tapanes et al., 2008). The *Jatropha Curcas* plant has been referred to as the second generation cropping solution for biofuel production (Gressel, 2008). Though the land requirement of third generation biofuel feedstock (for example, algae) is much less, the production of biofuel from these third generation feedstocks is much more complex than from the second generation feedstock (*Jatropha Curcas*). Moreover *Jatropha Curcas* grows in waste land, which makes it a more attractive feedstock for biodiesel production. *Jatropha curcas* is a drought-resistant oil bearing multipurpose shrub/small tree which belongs to the family of *Euphorbiaceae* (Staubmann et al., 1999; Ackom and Ertel, 2005; Achten et al., 2006). It originates from Central America and was distributed by Portuguese seafarers via the Cape Verde Islands to countries in Africa and Asia (Henning, 2000). These days jatropha is widely grown in Mexico, China, north-east Thailand, India, Nepal, Brazil, Ghana, Mali, Foso, Zimbabwe, Nigeria, Malawi, Zambia and some other countries (Ackom and Ertel, 2005; Openshaw, 2000). There are 175 species of jatropha around the world (Becker and Makkar, 2008). Jatropha grows in arid and semi arid climates and in a wide range of rainfall regimes, from 200 to 1500 mm per annum (Achten et al., 2006). It can survive in poor stony soils (Aderibigbe et al., 1997). The plant grows quickly forming a thick bushy fence in 6-9 months, up to a height of 4 m with thick branches in 2-3 years and the branches contain latex (Henning, 2000; Augustus et al., 2002). The life span of the *Jatropha curcas* plant is more than 50 years (Henning, 2000). Almost all parts of the plant have a medicinal value (Staubmann et al., 1999). The bark is rich in tannin and also yields a dark blue dye. The tender green leaves are fed to silk worms, for small scale silk production (Augustus et al., 2002). In many countries jatropha is planted in the form of hedges to protect gardens and field crops from roaming animals. Since jatropha plants have lateral roots near the surface, they can be used to fix small earth dams which reduce the flow of run-off water (Henning, 2000). Its seeds resemble castor seeds in shape, but are smaller and brown (Augustus et al., 2002) and have an annual seed yield of 5 tons per hectare (Aderibigbe et al., 1997). One estimation shows that the seeds contain 30-32% protein and 60-66% lipid (Augustus et al., 2002). The oil content of the seeds varies from 30 to 60% depending on the variety, place and the method of oil extraction. The seed and /or the oil have been found to be toxic, so the oil cannot be used for cooking purposes and the cake remaining after extraction of the oil from the seed cannot be used as cattle feed or for any edible purpose. The cake contains about 6% N, 3% P, and 1% K.  But the oil has an excellent fuel property.

This oil can be used for lighting and as fuel for cooking. It was used as diesel fuel substitute during World War II (Shah et al., 2004).

Biodiesel is a renewable diesel fuel, normally obtained by transesterification of vegetable oils, waste cooking oils and fats with lower alcohols (methanol, ethanol), in the presence of acid/base or enzyme catalysts at a lower temperature and pressure or in the absence of catalysts at a higher temperature and pressure. As the base catalyzed transesterification reaction is faster than the acid catalyzed reaction, most commercial processes use a homogeneous base, i.e. NaOH, KOH, or related alkoxides for biodiesel production (Ataya et al., 2007). Generally, methanol is used as it is cheaper than ethanol. One of the major problems with using traditional basic catalysts is the formation of soap when oils with high free fatty acids are used as feedstock. This results in lower yields of biodiesel and subsequent washing of biodiesel is required to remove the soap from the biodiesel to maintain its quality. When traditional basic catalysts (potassium or sodium hydroxide) is used as the catalyst, the hydroxide ion of these catalysts reacts with alcohol (i.e. methanol, ethanol), irrespective of whether the alcohol is anhydrous or not, and water is formed. Then this water reacts with triglycerides and as a result of a hydrolysis reaction, diglycerides and free fatty acids (FFA) are formed. This FFA reacts with potassium or sodium ions to form soap through a saponification reaction.

The main factor controlling the overall biodiesel production cost is the price of the refined oil feedstock (free of fatty acids), which accounts for 88% of the total estimated production cost (Di Serio et al., 2007). Thus many commercial processes use cheaper feedstock, i.e. waste cooking oil, waste fat and oils, despite their high free fatty acids content.

Hartman (1956) showed that potassium carbonate, a salt of potassium hydroxide, is better than traditional base catalysts (KOH, NaOH, NaOCH$_3$ and KOCH$_3$) as it ensures practically complete alcoholysis and produces the least amount of soap. But after his work, no one else tried using unsupported potassium carbonate as a catalyst for biodiesel production for a long time. One of the reasons may be the current potassium carbonate production technology. Among traditional catalysts, KOH and its methylate are the most expensive. At present KOH is produced by electrolysis of KCl solution. Then K$_2$CO$_3$ is produced by reacting KOH with CO$_2$. Thus the price of potassium carbonate produced chemically is higher than that of KOH.

In 1985, a group of French scientists used ashes of coconut shell and palm kernel as a catalyst for biodiesel production from coconut oil and palm oil respectively and obtained good results. The main component of those ashes was potassium carbonate (Graille et al., 1985; Graille, 1986). US patents 6890451 B2 (Sapienza et al,. 2005) and 7138071 B2 (Sapienza et al., 2006) showed that glycerol containing potassium carbonate can be an excellent environmentally

benign anti-icing or deicing fluid, which can be used on the wings, fuselage, and tail of aircrafts for de-icing, and in some instances on airport runways (Sapienza et al., 2005 and Sapienza et al., 2006).

Potassium is one of the major inorganic components of *Jatropha curcas* seedcake. The seedcake contains approximately 1% of potassium by weight (Openshaw, 2000), which can be recovered from the seedcake ash. *Jatropha curcas* oil contains free fatty acids which tend to form soap if base catalysts are used.

Thus there is the necessity for research to use reagent grade potassium carbonate as a catalyst for biodiesel production using jatropha oil because, among the base catalysts, potassium carbonate produces the least soap and, also, it is recoverable. The vision for using potassium carbonate as a catalyst in the present study is to recover it from the biomass of Jatropha curcas (for example, the ash from combustion of the seedcake after squeezing the oil), an approach that has the potential to benefit the economies of developing countries where Jatropha curcas can be grown on a large scale.

In recent years FTIR (Fourier transform infrared spectroscopy) has been tried as a method for detecting biodiesel conversion because of its easy and fast detection techniques. The raw oils and the methyl esters are fairly strong absorbers in the infrared region. From a cursory examination of the literature, there are commercial units that use an integration of the side $(1750 - 1760 \text{ cm}^{-1})$ of the carbonyl peak ($\sim 1744 \text{ cm}^{-1}$) to monitor the progress of the reaction. This method can be effective in a known system but is limited because it is not specific for the end product. There could be a number of interferences. The reason that the commercial units use this method is that they do not have to have spectral resolution to do more specific analysis. The peak typical of the methyl ester (O-$CH_3$) at $1436 \text{ cm}^{-1}$ is very narrow and rides on the side of another peak (oil). Both of these characteristics make this peak unattractive to the commercial units for monitoring the biodiesel reaction progress. But this peak measurement gives a direct indication of the attachment of the alkyl group of the alcohol with the fatty acids of the triglycerides and this peak is free of the influence of the alkyl group (-$CH_3$) present in the alcohol. Other FTIR-ATR analytical methods rely on the formation of glycerol as a side product rather than on the direct measurement of the formation of the methyl ester (monitoring methyl peak). Another advantage of using FTIR -ATR is the ability to detect the presence of soap in the biodiesel. Soaps are the sodium, potassium or other metal salts of the carboxylic (fatty) acids. In soap formation when the carboxyl groups (COO-) of fatty acids are attached to the metal ions, the $CO_2$ stretch band is usually seen at $1650\text{-}1540 \text{ cm}^{-1}$ (Lin-vien et al., 1991). This single peak is an indication of the presence of soap.

The purpose of the present study was to investigate the effect of catalyst amount, alcohol to oil molar ratio and reaction temperature on transesterification

of JTC. Specifically, the study focused on potassium carbonate as a catalyst, which is new because no previous study has looked at using potassium carbonate in JTC transesterification.

## 2. Materials and Methods

Anhydrous grade (99.995%) methanol was purchased from Alfa Aesar (Oakville, Ontario, Canada) and ACS grade (99%) $K_2CO_3$ was obtained from common chemical storage. Both triolein ($\approx$99%) and methyl oleate ($\approx$99%) were bought from Sigma Aldrich (Oakville, Ontario, Canada). Double press virgin *Jatropha Curcas* oil was purchased from Medors Biotech Pvt Ltd, New Delhi, India. The fatty acid composition (%) of the oil was (based on wt %).

Table 1. Composition of the raw jatropha oil

| Fatty acid Composition | |
|---|---|
| Palmitic acid (C16:0) | 15% |
| Stearic acid (C18:0) | 4.8% |
| Oleic acid (C18:1) | 27.22% |
| Linoleic acid (C18:2) | 32.48% |
| Linolenic acid (C18:3) | 3.41% |
| Arachidic acid (C20:0) | 1.1% |
| Eicosenic acid (C20:1) | 1.56% |
| Behenic acid (C22:0) | 0.78% |
| Lignoceric acid (C24:0) | 13.18% |
| Nervonic acid (C24:1) | 0.48% |

The free fatty acid content of the oil (wt% as oleic) was 2.86% and the peroxide value of the raw oil was 9.2 meq/kg.

Biodiesel (FAME) was synthesized in a glass batch reactor. The top of the reactor was fitted with a condenser to condense the methanol. Connected to the condenser was a U - shaped tube filled up with anhydrous calcium sulfate blocked by cotton on both sides of the tube to ensure entrance of moisture free air into the system. The Jatropha oil was stored in a cold and dark room at 5°C under argon to prevent oxidation. The oil container was flushed with argon after each opening. One hundred grams (110 mL) of jatropha oil were introduced into the reactor. Then 30 mL of methanol (6:1 methanol to oil molar ratio) and different amounts of catalyst were added to the reactor to examine the effect of catalyst amount. Following that, a fixed amount of catalyst (4 wt% of the oil) was kept and the methanol to oil molar ratio was varied while keeping the temperature (60°C) of the reaction constant. In experiments where the reaction temperature was varied,

6:1 methanol to oil molar ratio and 4 wt% catalyst (wt. based on the oil) were used. All the reactor contents were preheated to the desired temperature. Stirring was then started and this point was taken as the start of the reaction.

ASTM method D664 (ASTM 2009) was used to determine and compare the acid number of the biodiesel. The measured fatty acid methyl ester composition of the produced biodiesel was used to estimate the cetane number of the biodiesel using correlations developed by Bamgboye and Hansen (2008) and Gerpen (1996). The obtained cetane number was then compared to cetane numbers obtained from ASTM Standard D613 (ASTM 2008).

## 2.1 Calibration of the equipment

The sensitivity of the temperature sensing probe of the digital controller of the hot plate was checked using a traceable ISO 17025 calibrated lollipop digital thermometer (VWR, Oakville, Ontario, Canada) and the difference in temperature was $\pm$ 0.2 °C. The vendor specified a magnetic stirrer bar especially designed for this digital hot plate and stirrer. The stirring sensitivity with respect to the rpm was $\pm$ 2% as defined by the vendor. A 5-digit precision electronic balance was used to weigh the catalyst ($K_2CO_3$). The sensitivity of the balance was verified with reference weights. The weight indication was accurate up to 4 digits.

## 2.2 Sample preparation

Samples were withdrawn regularly from the reactor for analysis. A 1.5 mL reactor sample was withdrawn for each test and 9 mL of deionized water was added to wash out glycerol, methanol, catalyst and soap. The mixture was then allowed to settle for an hour. Next, a 1mL sample from the upper biodiesel phase was taken out and centrifuged at 6000 rpm for 30 minutes to remove the residual glycerol, methanol, catalyst and soap. Thus the samples were dried with anhydrous $Na_2SO_4$. Approximately, 300 μL of this sample was stored at - 18 °C before analysis. This sample is referred to as "Washed Sample". The rest of the reaction mixture in the reactor was poured into a separatory flask and the flask was then capped. After 14 hrs of settling a 2.5 mL sample of the upper phase (biodiesel) was taken out for analysis. The sample collected from the upper phase (biodiesel) is called "Unwashed sample". The analysis of the latter is important to understand the presence of soap in the actual biodiesel to assess its quality and the degree of washing needed to purify it. An additional 1.5 mL of the unwashed sample was taken and washed, following the same procedure used for the washed sample; this was referred to as "Washed sample after extended time (14 hours settling time)". This sample was analyzed to check the conversion of Jatropha oil to biodiesel and the change in the methyl ester concentration after settling.

## 2.3 Analysis of results

Bruker IFS 55 FTIR was used for analysis and a ZnSe through cell was used as the ATR (Attenuated Total Reflectance) cell. Air was taken as the background of the FTIR spectra. A 100 µL sample was placed on the ZnSe crystal plate. The IR light incident on the ZnSe crystal was at a $45^o$ angle, reflected off the crystal several times before leaving the crystal. All spectra were scanned 100 times and recorded at a resolution of 4 cm$^{-1}$. OPUS version 4.0 software was used to analyze the spectra in terms of absorbance mode, as quantitative analysis (area integration) is possible in this mode. Integration method B (baseline to base line) within that software was used to calculate the peak area. The progress of the transesterification reaction was monitored by measuring the FTIR area (1446 – 1428 cm$^{-1}$) under the methyl (O-CH$_3$) peak (1436 cm $^{-1}$), which accounted for the methyl esters of all types of fatty acids in the biodiesel. The presence of soap was qualitatively assessed by measuring the area of a single FTIR peak in the 1597 – 1544 cm$^{-1}$ range of the "unwashed sample" spectrum.

  The raw oil and the methyl esters are fairly strong absorbers in the infrared region (Fig. 2) and precaution was taken to ensure that the spectra were not saturated when performing the analysis. Saturated spectra occur when none of the IR light is transmitted at a particular wavelength and all the light is absorbed. Under this circumstance, it is impossible to make a quantitative analysis based on the peak. In saturated spectra, the peaks either have the same height or appear to be "grassy".

  For quantitative measurements of the reaction progress, different biodiesel standard solutions were prepared using triolein and methyl oleate by mixing them in different molar concentrations (Fig. 3). The raw oil was assimilated to the glycerol ester of oleic acid (triolein) and the biodiesel was assimilated to the methyl oleate. These standard solutions were used to prepare a calibration curve which followed a second order polynomial form.

$$X = - 16.474a^2 + 90.725a - 25.153 \tag{1}$$

where X = mole concentration of FAME; a = area under O-CH$_3$ peak.

  The probe depth of the ATR crystal is dependent on the refractive index of the sample which is dependent on the viscosity of the sample. The viscosities of the beginning material and of the end product are not the same. Moreover, the FTIR response is also dependent on the extinction coefficients of the material in the sample and the coefficients are probably not the same for the raw oil and the biodiesel product (personal communication with M. J. Waljak). These spectrometer for biodiesel analysis (http://www.wilksir.com/pdf/App-note_Biofuel-InfraSpec.pdf. ).
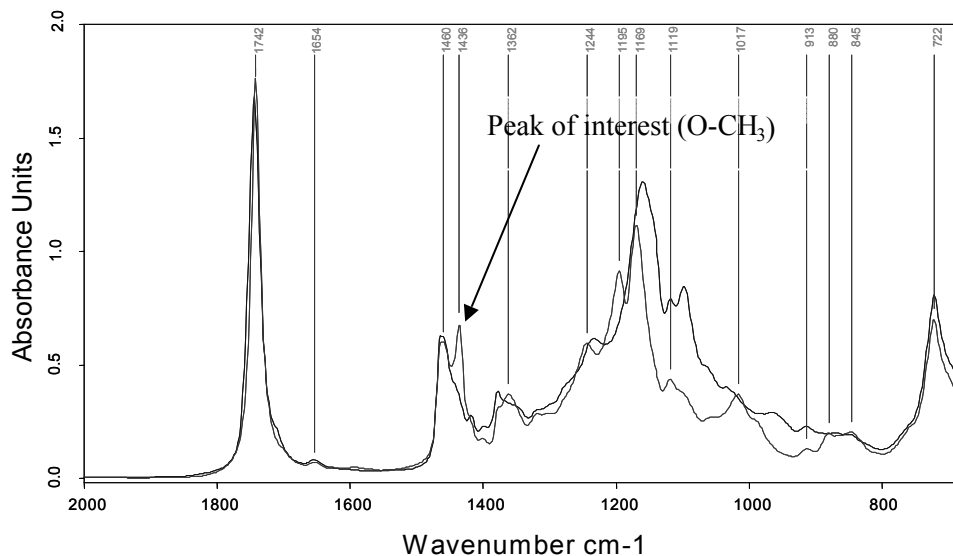
Figure 2. FTIR - ATR Spectra of raw jatropha oil and pure biodiesel produced from jatropha (JTC) oil.
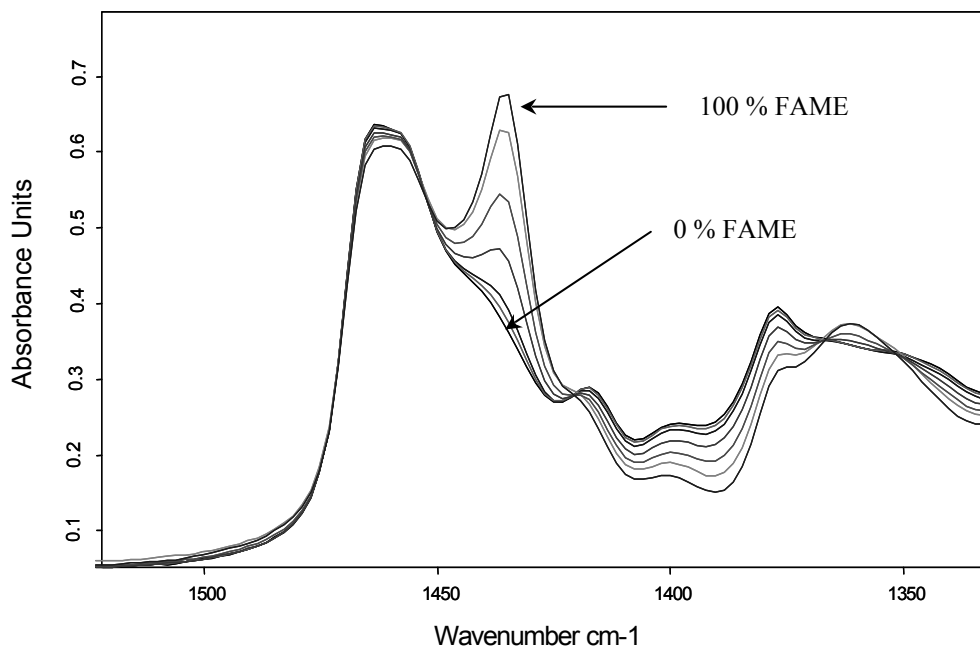


Figure 3. Change in methyl peak (O-CH$_3$) with molar concentration of FAME in the standard solution

The mole concentration (mole % or mole fraction) of FAME (fatty acid methyl ester) in the reaction sample obtained using the calibration curve was used to calculate the % mole conversion in the following way. If "X" is the mole fraction of FAME (biodiesel) present in the sample then,

$$\% \text{ mole conversion} = \frac{\dfrac{x}{3}}{\dfrac{x}{3} + (1-x)} \times 100 \% \tag{2}$$

The factor $\dfrac{1}{3}$ in Equation (2) arises from the stoichiometry of the transesterification reaction (Fig.1).

## 3. Results and discussion

### 3.1 Effect of catalyst amount

It is observed from Fig. 4 that with a higher catalyst amount, a greater mole percent conversion in the reaction was achieved within a shorter reaction time. However, after one hour the percent mole conversion stabilized for almost all catalyst amounts except when 2 wt% (based on wt of oil) catalyst amount was used. This indicates that 2 wt% potassium carbonate is not sufficient to catalyze the transesterification reaction, for one hour of reaction time.
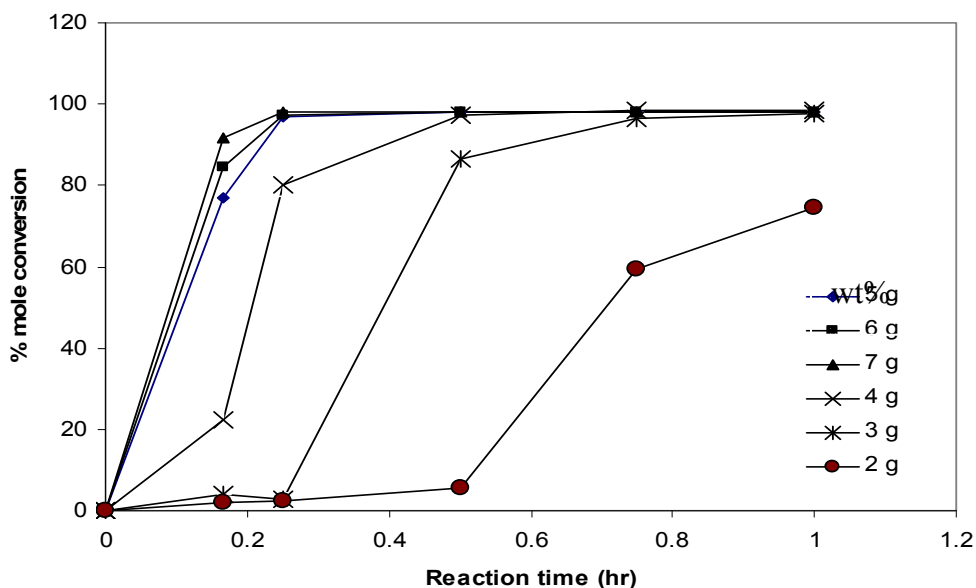


Figure 4. Effect of catalyst amount (60°C, 6:1 methanol to oil molar ratio)

In separate runs when the transesterification reaction was allowed to run for 10 hours using different amounts of catalyst, the results show that 6 percent potassium carbonate (wt% of jatropha oil) gave the highest mole conversion (98.214%), when 100 g of jatropha oil was reacted with methanol at a 6:1 methanol to oil molar ratio at a temperature of 60°C with stirring at 600 rpm for 10 hours (Table 2). The change in percent mole conversion was further checked by determining the conversion for the unwashed sample. It was found that the difference in percent mole conversion between washed and unwashed samples increased with an increase in the catalyst wt% (Tables 2 and 3).

Table 2. Effect of catalyst amount on percent mole conversion of the reaction (washed sample) after 10 hours

| Catalyst amount (wt% of jatropha oil) | Washed sample | |
| --- | --- | --- |
| | Average % mole conversion of the raw oil | Error% at 95% confidence level |
| 1 | 2.51 | 1.63 |
| 2 | 92.79 | 0.37 |
| 3 | 94.95 | 1.08 |
| 4 | 96.84 | 0.28 |
| 5 | 97.85 | 0.64 |
| 6 | 98.21 | 0.12 |
| 7 | 97.48 | 0.39 |

Table 3. Effect of catalyst amount on the % conversion of the reaction (unwashed sample) after 10 hours

| Catalyst amount (wt% of jatropha oil) | Unwashed sample | |
| --- | --- | --- |
| | Average % mole conversion of the raw oil | Error% at 95% confidence level |
| 1 | - | - |
| 2 | 91.34 | 1.57 |
| 3 | 94.75 | 0.57 |
| 4 | 92.50 | 1.79 |
| 5 | 90.08 | 2.85 |
| 6 | 89.29 | 1.18 |
| 7 | 65.87 | 5.80 |

It was found that the difference in percent mole conversion between washed and unwashed samples increased with an increase in the catalyst wt% (Tables 2 and 3).

The amount of soap was estimated in the unwashed sample by measuring the area of the peak in the wave number range $1597 - 1544$ cm$^{-1}$. The results showed that the amount of soap formed was almost the same for 2-3 wt% catalyst but increased from 3% catalyst (wt% of jatropha oil) (Fig. 5). Figure 5 shows further that the amount of soap was almost the same again for $5 - 6$ wt % catalyst. The amount of soap increased dramatically when 7 wt% catalyst was used. This was an indication of saponification of triglycerides as a secondary reaction with respect to transesterification. Previous studies showed that excess alkali catalyst caused the saponification of triglycerides resulting in the formation of soap and in an increase in the viscosity of the reactants. It caused lower ester formation (Dorado et al., 2004; Rashid and Anwar, 2008). Thus secondary saponification might be responsible for significantly lower conversion of jatropha oil to biodiesel (FAME) when 7 wt% potassium carbonate catalyst was used instead of 6 wt%. Figure 5 also indicates that the amount of soap formation increased with time when excess amount of catalyst (7 wt %) was used for an extended time (10 hours).

When 1 wt% catalyst was used, the reaction intermediate formed was highly unstable in the presence of air and it was difficult to get repeatable results using FTIR-ATR. This was an indication that 1 wt% catalyst was not enough to catalyze the reaction to produce FAME.

The decrease in percent mole conversion for the unwashed sample was not due to any FAME (biodiesel) degradation. This was confirmed by checking the percent molar conversion of the washed sample after an extended time (14 hours settling time of the reactor contents). For 2 and 7 wt% catalyst, the percent molar conversion of the washed sample (before settling) and of the washed sample after extended time (14 hours settling time of the reactor contents) was almost the same, as shown in  Table 4.

Table 4. Effect of catalyst amount on percent mole conversion of the reaction (washed sample after extended time: 14 hours of settling) for 10 hours reaction time

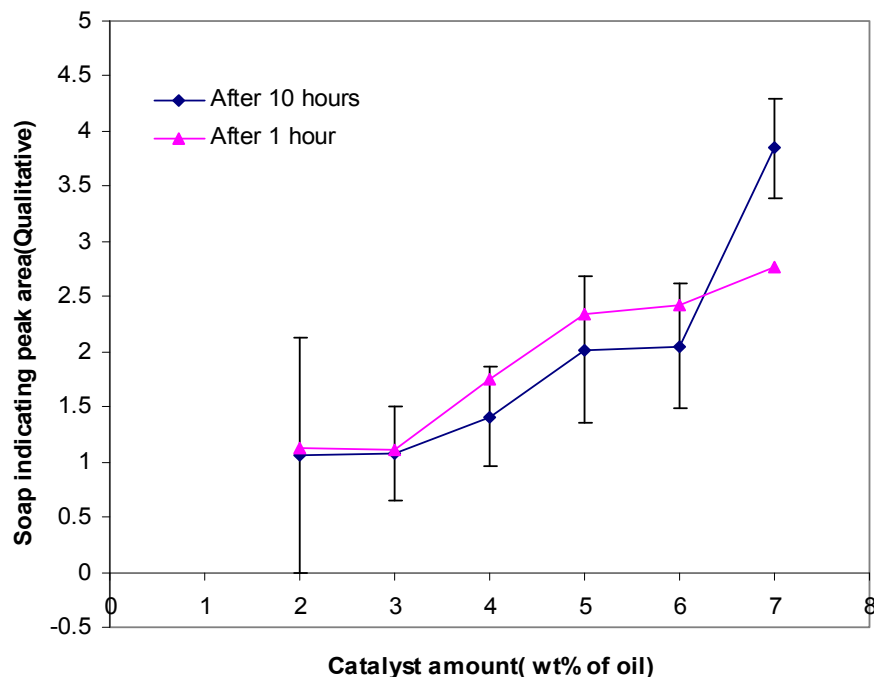| Catalyst amount (wt% of jatropha oil) | Washed sample after extended time ( 14 hours of settling) | |
|---|---|---|
| | Average %  mole conversion of the raw oil | Error% at 95% confidence level |
| 2 | 93.12 | 0.78 |
| 7 | 97.60 | 0.15 |

Figure 5. Effect of catalyst amount on the by-product soap formation (60°C, 6:1 methanol to oil molar ratio) using unwashed sample

The decrease in percent molar conversion at high catalyst amount was due to the reduction in the molar concentration of FAME in the biodiesel phase which resulted probably from the increased solubilization of soap and other components into the biodiesel phase. The qualitative soap presence (Fig. 5) also agreed with this conclusion. These results confirmed that the saponification reaction occurred during the transesterifcation reaction and that there were no side reactions during the settling of the biodiesel.

A previous study on the solubility of anhydrous potassium carbonate in methanol showed that potassium carbonate and methanol undergo a reversible reaction in the following way (Platonov et al., 2002):

$$CH_3OH + K_2CO_3 \rightleftharpoons CH_3OK + KHCO_3 \qquad (3)$$
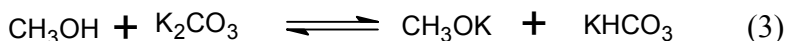
That study showed that more than 99% of the total quantity of $KHCO_3$ generated in the above reaction remained in the solid phase along with unreacted potassium carbonate at room temperature (25°C). The phase distribution of $KHCO_3$ between solid and liquid phase promoted the shifting of the equilibrium of the reaction towards product formation according to Le Chatelier's principle. Increasing the temperature caused more $KHCO_3$ to dissolve into the liquid phase from the solid phase. This increased the rate of the reverse reaction. As a result,

the concentration of CH$_3$OK decreased and, at the same time, the concentration of K$_2$CO$_3$ increased (Platonov et al., 2002). However, KHCO$_3$ was found to be a poor catalyst with negligible catalytic activity when compared to that of K$_2$CO$_3$ (Arzamendi et al., 2008). This indicates that CH$_3$OK formed from the reaction between K$_2$CO$_3$ and CH$_3$OH is the main catalyst compound. Thus the result of the transesterification reaction is largely dependent on the CH$_3$OK concentration and hence on the reaction in equation (3).

## 3.2 Effect of methanol to oil molar ratio

Since excess amount of catalyst produces excess soap over time and yields lower conversion, it is necessary to obtain a higher conversion within the shortest possible time using a low amount of catalyst. For this, a fixed amount (4 wt% on oil) catalyst was selected and the methanol to oil molar ratio was varied from 6:1 to 11:1 and the results are shown in Fig. 6.
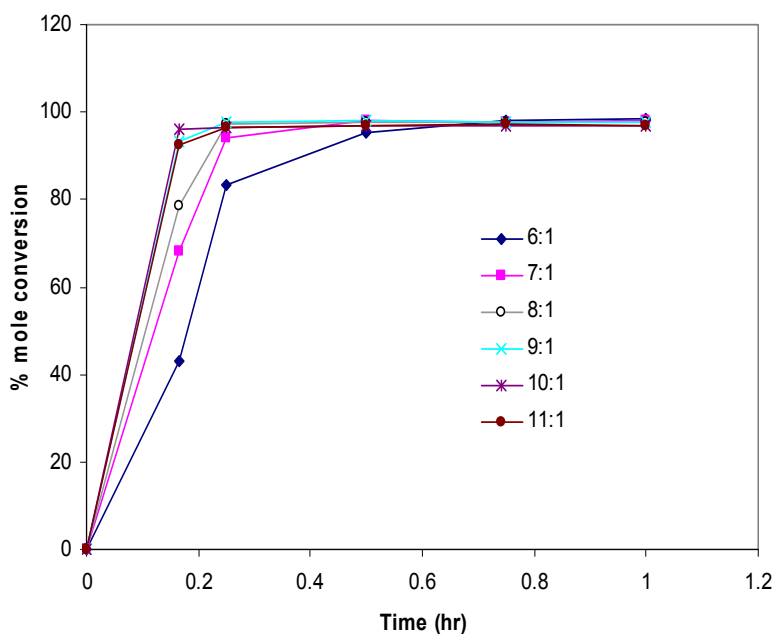


Figure. 6 Effect of methanol to oil molar ratio (4 wt% catalysts, 60°C)

The results showed that with the increase in the methanol to oil molar ratio, the percent molar conversion increased rapidly with time but after 1 hour the ultimate percent mole conversion was lower compared to that of using lower methanol to oil molar ratio. This showed that the methanol to oil molar ratio had

an effect on the initial higher conversion of the oil to biodiesel but little or no effect at the later stage in the reaction.
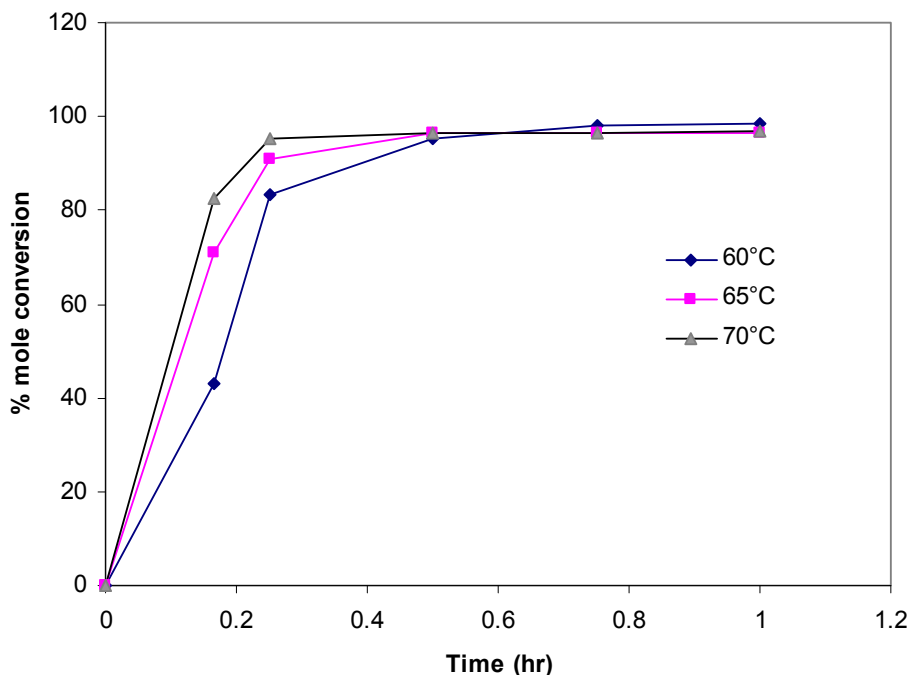
## 3.3 Effect of reaction temperature



Figure 7. Effect of reaction temperature (4 wt% catalyst, 6:1 methanol to oil molar ratio)

Figure 7 shows that with an increase in the reaction temperature at or above the boiling point of methanol (65°C), the initial reaction conversion rate increased rapidly but after 1 hour, the ultimate percent mole conversion appears to be lower compared to that of using a lower temperature. The probable reason for this is that above the boiling point, methanol loss by evaporation resulted in less methanol available in the liquid phase for the transesterification reaction, which led to lower conversions. Some studies have demonstrated that the saponification of glycerides by the alkali catalysts is much faster than the transesterification reaction at temperature above 60°C (Patil and Deng, 2009; Eevera et al., 2009). In other words, higher temperatures than 55°C accelerates the side saponification reaction of the glycerides ( Eevera et al., 2009). This may be also the reason for the observed lower conversion at the reaction temperature of 65°C in the present study.  In order to minimize methanol loss at a temperature higher than 60°C, we would have had to build a unit that could be pressurized to condense the methanol vapour and reflux it to the reaction zone. Our study did not explore this option.

Thus operating the reactor under the conditions of the experiments at temperatures higher than 60°C was not economical.

### 3.4 Quality of the biodiesel

For a typical parameter combination of 6 wt % catalyst, 6:1 methanol to oil molar ratio, 60°C and 10 hours reaction time, the quality of the biodiesel was analyzed. The compositions of the biodiesel are shown in a tabular form.

Table 5. Composition of the biodiesel (6 wt % catalyst, 6:1 methanol to oil molar ratio, 60°C and 10 hours reaction time)

| Composition of biodiesel | |
|---|---|
| Palmitic acid methyl ester | 16.68% |
| Stearic acid methyl ester | 6.4% |
| Oleic acid methyl ester | 51.81% |
| Erucic acid methyl ester | 14.00% |
| Other fatty acid methyl ester | 3.9% |
| Free glycerol | 0.034% |
| Monoglyceride | 0.0762% |
| Other organic compaounds | 7.09% |

The acid number of the produced biodiesel was 0.54 mg KOH/g, whereas ASTM specified acid number should be 50 mg KOH/g. The viscosity at room temperature (25°C) was 5.97 cP. The fatty acid methyl ester composition of the produced biodiesel was used to predict the cetane number of the produced biodiesel using different correlations (Bamgboye and Hansen, 2008; Gerpen, 1996) as the conditions of these correlations were similar to the one here. The predicted cetane numbers using different correlations were 50.4, 50.61 and 53.19, which were very close. The total glycerin (free glycerin + monoglycerides) found in the biodiesel composition was also in the tolerable limit (0.24 wt% max).

## 4. Conclusions

*Jatropha Curcas* oil can be an alternative feedstock for biodiesel production and can reduce the demand on food crops for renewable fuel production. In previous biodiesel production experiments from various feedstocks, potassium carbonate was used as a supported catalyst. In the present study, it was used as an unsupported catalyst to understand its catalytic activity in the transesterification reaction. Based on the results, the following is concluded:

- A 3 wt% potassium carbonate was shown for the first time to be sufficient to complete the transesterification of Jatropha oil within 1 hour of the start of the reaction, when 6:1 methanol to oil molar ratio and 60°C were used.

- When the amount of potassium carbonate was increased to 5 wt%, transesterification was complete within 15 minutes at 6:1 methanol to oil molar ratio and 60°C.

- Excess amount (say 7 wt %) of catalyst saponified the triglycerides and this saponification appeared to increase with reaction time.

- Higher reaction temperature had an effect on the initial conversion rate, but the ultimate conversion was lower compared to that of using a lower reaction temperature.

Potassium carbonate has a great potential as a catalyst for the transesterification of JTC. Every biomass ash contains this mineral in a significant amount. Potassium carbonate can be easily extracted from biomass ash using inexpensive classical extraction technology.

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