## THE INFLUENCE OF ULTRASONIC AMPLITUDE AND TEMPERATURE ON TRANSESTERIFICATION OF JATROPHA CURCAS OIL

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**Abstract** Transesterification of Jatropha oil with methanol was carried out in the presence of sodium hydroxide catalyst. Ultrasonic energy from Ultrasonic Processor (UP50H) was used to induce the transesterification of the oil. The ultrasonic processor has a frequency of 30 kHz and power of 50W. The influence of the ultrasonic amplitude and temperature of reaction using UP50H was investigated on the yield of biodiesel from jatropha oil. The maximum obtainable amplitude of the device is 125 $\mu$ m and the amplitude was varied from 60 to 90 % of the maximum. The influence of reaction temperature was considered from 40 to 60 °c at a catalyst concentration of 0.5 %w/w of oil and a reaction time of 30 minutes. The highest yield of 94.44% obtained from this work was achieved at amplitude of 100  $\mu$ m (80%) and a temperature of 50°c for three mole ratio used. The biodiesel obtained was comparable to diesel standard.

Keywords: ultrasonic, amplitude, temperature, transesterification, biodiesel, yield

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**1.Introduction** Biodiesel has drawn significant attention due to increasing environmental concern and diminishing petroleum reserves. Some of the advantages of using biodiesel fuel are renewable, non toxicity and safer handling due to its higher flash point compared to those of fossil fuel (Wang., et al

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 Department of Chemical Engineering, University of Lagos, Lagos 2006). Biodiesel fuel primarily contains no sulphur and aromatics, producing better gas exhaust emission than conventional fossil diesel fuel (Demirbas, 2009). Biodiesel is an alternative fuel produced from renewable vegetable oils, animal fats or recycled cooking oils by transesterification of triglycerides(TG) with alcohol, commonly methanol, in the presence of a base or acid catalyst into fatty acid methyl esters (FAME). There are likely three stepwise reactions that occur with the formation of the diglycerides (DG) and monoglycerides (MG) intermediates. This results in the production of 3 mol of FAME and 1 mol of glycerine (GL) from 1 mol of TG, as shown in the mechanism below (Damoko and Chervan, 2000).

The overall reaction is:

#### catalyst

TG + 3CH<sub>3</sub>OH 
$$\leftarrow$$
 GL +3FAME  
The stepwise reaction are  
TG + 3CH<sub>3</sub>OH  $\leftarrow$  DG+FAME 1  
DG + 3CH<sub>3</sub>OH  $\leftarrow$  MG+FAME 2  
MG + 3CH<sub>3</sub>OH  $\leftarrow$  GL+FAME 3

The high energy demand in the industrialized world as well as in the domestic sector, and pollution problems caused due to the widespread use of fossil fuels make it increasingly necessary to develop the renewable energy sources of limitless duration and environmentally friendly. These have stimulated recent interest in alternative sources for petroleum-based fuels (Rahman., et al , 2010). Many developed countries have active biodiesel programs. Currently biodiesel is produced mainly from field crop oil like rapeseed,

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 Department of Chemical Engineering, University of Lagos, Lagos sunflower etc. in Europe and soybean in US. Malaysia utilizes palm oil for biodiesel production while in Nicaragua it is jatropha oil. There are many countries which have large amount of biodiesel potential. If this potential is used for the production of biodiesel then the crisis of petroleum based diesel and fossil fuel can be solved. The global warming problem can also be solved because biodiesel is bio- fuel and it has no harmful emission in diesel engine. One way of reducing the production costs for biodiesel fuel is the use of non edible oils, which tend to be considerably cheaper than edible vegetable oils. Jatropha Curcas oil cannot be used for food purposes, because of it toxic nature. Jatropha curcas plant is adaptable to large variety of soils, altitude and rain volume, the plant yields seed oil rich in oleic and linoleic acid (Haas, et al., 2002). Hence, many scientists are interested in exploring the potential values of this plant. Now a day's various techniques have been developed for producing biodiesel. Some of which are mechanical stirring, ultrasonic cavitation, hydrodynamic cavitation and supercritical methanol. Batch reactor with mechanical stirrer have been the practice but this conventional approach the to transesterification of triglycerides to methyl ester is slow and low yield. The need and importance of Ultrasonic processor was introduced to eliminate mass transfer resistance as it ensures better mixing and consequently lead to better yield. Ultrasonication processor technique is a new and promising technology. Ultrasonic cavitational mixing is an effective alternative means to achieve a better mixing in transesterification reaction. Sonochemistry is the application of ultrasound to chemical reactions processes. The mechanism and causing sonochemical effects in liquids is the phenomenon of acoustic cavitation. Cavitations can be produced in ultrasonic transducers. Ultrasonic traducers use electric excitation with a frequency of 30 kHz to generate mechanical vibrations, which is transferred to the liquid medium through sonotrode. The sonotrode is made of titanium alloy. A titanium alloy has proven to be the best sonotrode material as it allows very high amplitudes and as it is resistant to most liquids. Therefore this titanium alloy is the standard material for sonotrodes and the ultrasonic processors are adjusted to it. The ultrasonic processor UP50H (50 watts, 30kHz) is the smallest model. The power output of the processor can be adjusted between 20 and 100 % of the maximum output. The maximum amplitude is 125µm - 220 µm depending on the sonotrode. The sonotrode for UP50H is MS7

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 Department of Chemical Engineering, University of Lagos, Lagos Micro tip 7 with a maximum amplitude of 125 µm and the maximum submerged depth is 30 mm. The structure of the ultrasonic processor (UP50H) is shown in Figure 1, while the operating and display elements of the ultrasonic processor is shown in Figure 2 respectively. Ultrasonic device has the following advantages :It will improve chemical reaction kinetics and consequently quality and yield is enhanced, It provides better mixing, less excess methanol is used; a molar ratio between 1:4or 1:4.5 (oil: methanol) is sufficient for most feedstock, when using ultrasonic mixing; ultrasonic mixing improves the methanol-in-oil emulsification and generates more and smaller droplets, which result in better distribution and increased catalyst efficiency; cavitations ultrasonic eliminate/improves the mass-transfer resistance in biodiesel production; it takes less time and consume less power than mechanical stirring technique and lower process temperature.

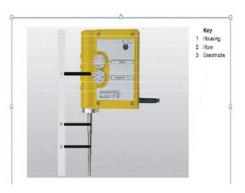


Figure 1:Structure of the Ultrasonic processor

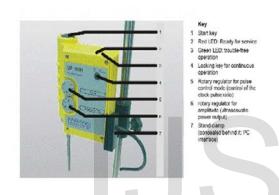


Figure 2: Operating and display element of the ultrasonic processor.

Larpkiattawom *et al.*, (2010) studied the effect of ultrasonication reaction of jatropha oil to biodiesel in both homogeneous and heterogeneous catalyst systems. They obtained biodiesel with methyl ester content of up to 97-98 % .Abhishek, (2009) studied biodiesel production through ultrasonic cavitation process and performance testing.

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 Department of Chemical Engineering, University of Lagos, Lagos He produced biodiesel from jatropha oil, thumba oil, and waste cooking oil and carried performance testing on 4-sroke,4-cynlinder water cooled Tata indica C.I engine. He reported that the result were in favour of biodiesel over diesel oil. Thanh, et al.,(2009) studied biodiesel production from canola oil with methanol in the presence of a basecatalyst by ultrasonic irradiation of low frequency (20 kHz) with an input capacity of 1 kW. Their optimal conditions were: methanol/oil molar ratio of 5:1 and 0.7 wt.% catalyst in oil and obtained the conversion of triglycerides to fatty acid methyl esters was greater than 99% within the reaction time of 50 min. The objective of this work is to investigate the influence of ultrasonic processor amplitude and temperature on the production of biodiesel from jatropha curcas oil.

### 2.Experimntal Method

#### 2.1 Materials and Apparatus

#### 2.2 Procedure

250g of the Jatropha oil was measured and charged into the beaker. Methanol to Jatropha oil molar ratio of 4:1,5:1 and 6:1were used respectively. The beaker containing the Jatropha oil was placed in a water bath and the water bath was set to the desired working temperature of 40°,50°,55° and 60° C respectively. The system was allowed to attain the desired temperature which was confirmed by using a thermometer. The sodium hydroxide 0.5 %w/w of oil was dissolved in methanol in another beaker and placed in the water bathe to attain the desired temperature. The sonotrode of the ultrasonic device was inserted into the beaker containing the Jatropha oil already in the water bath to attain the temperature. The ultrasonic device is then switched on and the mixture of methanol and sodium hydroxide was poured into the oil simultaneously. The knob was used to varied the amplitude to the working percentage (60-90 %). The reaction was timed as soon as the ultrasonic device was switched on. At the end of 30 minutes the ultrasonic device was switched off and the reactor mixture was emptied into a separating funnel; the resulting mixture in the separating funnel was kept to settle and the glycerol which is the heavier component was obtained first through the bottom of the separating funnel. The procedure was repeated with fresh jatropha curcas oil at the various conditions reported in this work. The

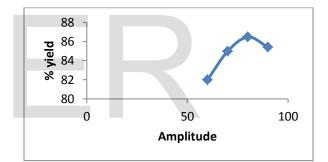
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 Department of Chemical Engineering, University of Lagos, Lagos biodiesel produced was characterize for some of its properties

#### 3. Results and Discussion

#### 3.1 Results of Experiment

Table 1: Effect of amplitude on yield attemperature 40 °C, catalyst concentration0.5%w/w, and time 30 min

| Amp   | %yield | %yield | %yield |
|-------|--------|--------|--------|
| (μm)  | 6:1    | 5:1    | 4:1    |
| 75    | 82.00  | 80.50  | 79.65  |
| 87.5  | 84.98  | 85.01  | 85.03  |
| 100   | 86.47  | 86.50  | 87.13  |
| 112.5 | 85.41  | 85.37  | 86.0   |



**Figure 3**: Effect of amplitude on yield at mole ratio 6:1 at temperature 40°C, catalyst concentration 0.5 %w/w and time 30 min

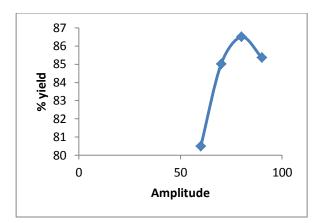


Figure 4: Effect of amplitude on yield at mole <sup>0</sup>C,catalyst ratio 5:1 at temperature 40 concentration 0.5 %w/w and time 30 min

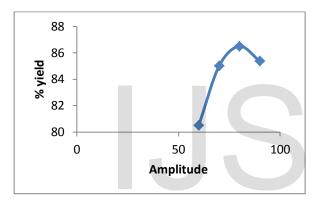


Figure 5: Effect of amplitude on yield at mole ratio 4:1 at temperature 40 °C,catalyst concentration 0.5 %w/w and time 30 min

Table 2: Effect of Temperature on Yield at Amplitude 80% (100 Catalyst μm), concentration 0.5 %w/w, Time 30 min

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| Temp | %yield | %yied | %yield |
|------|--------|-------|--------|
| (°C) | 6:1    | 5:1   | 4:1    |
| 40   | 86.13  | 86.70 | 87.11  |
| 50   | 93.63  | 94.01 | 94.44  |
| 55   | 90.11  | 90.43 | 91.00  |
| 60   | 88.27  | 88.67 | 89.20  |

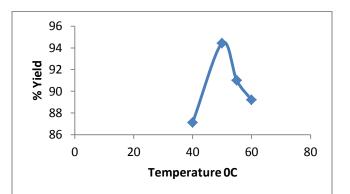
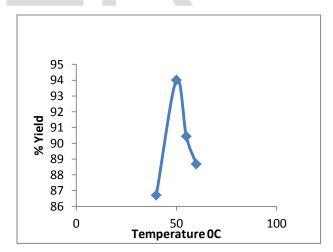


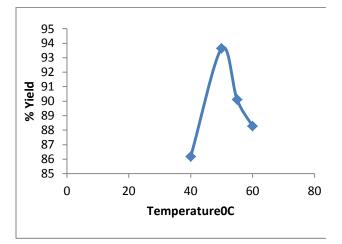
Figure 6: Effect of temperature on yield at amplitude 80% (100 µm), catalyst concentration 0.5 %w/w, time 30 min at mole ratio 4:1



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Figure 7: Effect of temperature on yield at amplitude 80% (100 µm), catalyst concentration 0.5 %w/w, time 30 min at mole ratio 5:1



**Figure 8**: Effect of temperature on yield at amplitude 80% (100 μm), catalyst concentration0.5%w/w,time30min,moleratio6:

 Table 3: Fuel characterization result of jatropha biodiesel

| <b>Biodiesel properties</b> | Jatropha oil | ASTM      |
|-----------------------------|--------------|-----------|
|                             | biodiesel    | standard  |
| Specific gravity at         | 0.883        | 0.87-0.89 |
| 30°C                        |              |           |
| Kinematic viscosity         | 4.78         | 3.7-5.8   |
| at 40°C (cm³/s)             |              |           |
| Cloud point °C              | 7            | -11 to 16 |
| Pour point °C               | 3            | -15 to 13 |
| Flash point °C              | 177          | 174       |

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<sup>b&c</sup> Prof. of Chemical Engr.Department of Chemical Engineering, University of Lagos, Lagos The use of ultrasonic processor (UP50H) for the transestrification of jatropha oil has been considered. The key factors considered in this work are the of amplitude ( intensity of stirring) and temperature when using ultrasonication method at three different mole ratios.

The effect of ultrasonic amplitude on transesterication of jatropha oil is shown in Figures 3,4 and 5. Effect of ultrasonic power on biodiesel yield was investigated with methanol to Jatropha-oil molar ratio of at 0.5 wt% catalyst and 4:1,5:1 and 6:1 temperature of 40°C. Biodiesel yield was highest at ultrasonic amplitude of 80%(100µm) in all the mole ratios. The yield increased slightly as ultrasonic amplitude increased from 75 to 100 µm, but drop slightly when amplitude was increase to 90%(112.5 µm) in all cases. The yield at amplitude 100 µm and mole ratio of 4:1 was 87.13 % maximum (Figure 4).The approximate yield at amplitude of 75 µm was 80 %, which increased to 87% as amplitude was increased to 100 µm and subsequently a decreased to approximate yield of 85% at amplitude of 112.5 µm. The optimal amplitude therefore from this work is 100 µm. Ultrasonic irradiation promotes sufficient mixing, helped in the formation and collapse of micro-scale bubbles to generate local high temperature and high pressure, and also provided alternative energy source to promote reactions ( Hingu .,et al ,2010). However, as the ultrasonic amplitude increased further from 80% (100 μm) to 90 %(112.5 μm) biodiesel yield dropped from 87 to 85 %. The possible reason could be that methanol was vaporized at higher ultrasonic amplitude. The experiment was conducted for 30 minute based on the result of previous study (Oghome ,2013).

In Figures 6,7 and 8, the effect of reaction temperature are represented at the various

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mole ratios considered in this work. Temperature has a paramount effect on yield and conversion. When ultrasonic device is used, energy required is less compared to other methods of production. From the results obtained ,it was observed that 50 °C was the optimum temperature. In all the mole ratios, 50 ° C gave the highest yield. When mole ratio was increase from 4:1 to 5:1 and 6:1, the optimum temperature was still the same but yield dropped because of the influence of other factors but remained highest at 50 °C. Deng, et.al., 2011 considered the influence of temperature on biodiesel yield using ultrasonic device. They conducted the experiment with 1wt% catalyst and 4:1 molar ratio of methanol to oil. They obtained optimum biodiesel yield 94.2% at optimum temperature of 45°C in 1.5 hours. They observed that yield decreased as temperature increased further. They reported that when temperature was higher than 45°C, methanol would vaporize and form a lot of bubbles, which could inhibit reaction. Abhishek (2009), reported a temperature range of 40-55°C in his study with ultrasonic device. Seung et al.,2011 reported that methyl ester content increased with increasing reaction temperature, but slightly decreased at the temperature higher than 55 °C. This phenomena can be explained as evaporation of methanol due to the boiling temperature of methanol of 64.7 °C, which results in its above 55 °C and decreases the possibility of the vegetable oil molecules coming into contact with it.

#### 4 Conclusion

In this experimental investigation, it was confirmed that the use of ultrasonic processor (UP50H) is a promising device for the production of biodiesel from Jatropha oil. The effect of temperature and amplitude on the yield at 30 min reaction time, and catalyst 0.5 %w/w concentration oil on was analyzed. The best and optimal reaction temperature was50 °C; amplitude of 100um at 4:1 molar ratio of methanol to oil. At these conditions the yield of biodiesel was 94.44 %. The result confirmed that ultrasonication technique enhances biodiesel yield and has efficiency of above 80 %.Biodiesel an characteristics like density, viscosity, flashpoint, cloud point and pour point are comparable to biodiesel standard

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