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Production and analysis of biodiesel from *Jatropha curcas* seed

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ABSTRACT: This present reported work was conducted to extract oil from *Jatropha curcas* seed, followed with the production of biodiesel via transesterification of resultant oil. The effects of methanol-to-oil ratio 1:1, 2:1, 3:1, 4:1, 5:1 and 6:1, reaction time of 30, 60, 90, 120 and 180min, at constant operating temperature of 60^{o}C were investigated. Also the energy input required for production of a unit biodiesel was calculated. The results of the study shows maximum biodiesel yield of 86wt% at methanol-to-oil ratio of 6:1, at reaction time of 180min. An energy input of ~1.4MJ/kg was estimated to produce a unit biodiesel from *Jatropha curcas.* The data reported in this study would improve fundamental knowledge in biodiesel production from *Jatropha curcas* seed as a renewable energy to complement fossil fuel.

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Growth in population and industrialization has led to an increased in energy demand worldwide. For decades fossil fuels have been the major supply of energy, of which about 90% being consumed as liquid fuel for transportation and energy generation (Devanesan *et al*., 2010). In addition, fossil fuel are non-renewable and have led to global warming, and environmental pollution. As a result of these negative environmental impact, dwindling reserves has led to researches on technologies and sources of alternative renewable energy that complement fossil fuels.

A number of technologies ranging from biochemical to thermochemical processes for liquid fuel renewable energy production have been reported in the literature. These processes employed biomass as feedstock for renewable energy production. Hence there has been an increase research on diverse feedstocks amongst which is biomass, Review of the literature shows liquid fuel production from lignocellulosic biomass, however, these biomass sources do compete with the food chain. It is important to state that such feedstocks should be non-competitive with the food chain.

Biodiesel-a biochemical process is one of the renewable and sustainable alternative liquid fuel that could complement fossil fuel. Biodiesel was found to be an alternative to fossil fuel primary because of its similar characteristics as diesel obtained from biomass can be applied directly in diesel engines without prior modification to achieve similar efficiency. In addition, biodiesel produced from vegetable oils has higher octane number, better lubrication properties, possesses both lower emissions and higher flash point, lower sulfur content, Also, they have lower aromatic content, biodegradability and renewability when compared to fossil diesel (Oladimeji and Oyekunle, 2015). However, its draw backs include lower volatility, reactivity of unsaturated hydrocarbons and are highly viscous (Demirbas, 2003).

Several feedstocks for biodiesel has been considered in the past. They include mostly vegetable oils such as kusum oil, soybean, palm oil, sunflower, rapeseed and coconut oil (Freedman *et al*., 1986; Noureddin and Zhu, 1997; Oladimeji and Oyekunle, 2015; Sanjel *et al*., 2014). However, due to issues of competition with food chain, the use oils extracted from microalgae, bacteria and fungi has also been investigated (Amin, 2009, Demirbas, 2009, Huang, 2010) within the last decade. However, feedstocks with high fat or oil content are preferred for biodiesel production. One of such feedstocks is *Jatropha curcas* seed, a drought resistant plant with over 30years life span of productivity. The plant has an oil content in the range of 25% to 60% (Deng *et al*., 2011), a significant amount suitable for biodiesel production. Importantly, it has similar fatty acid composition with edible oil, though, it contains toxic substances such as curcin, rendering it unsuitable for consumption (Openshaw, 2000; Tamlampudi *et al*., 2007). Therefore a non-competing feedstock with the food chain for biodiesel

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production. Gui *et al*., (2008) reported that about 95% of biodiesel is still being produced from edible oils which are tagged as competitive with food chain. In addition the use of edible oils for biodiesel production in developing countries like Nigeria is uneconomical as it would affect the food chain, hence producing biodiesel from non-edible oils should be encouraged. Interestingly, *Jatropha curcas* is among nonedible oils currently found in Nigeria, and they are economically un-useful and invaluable (Aranisola *et al*., 2012). For biodiesel production from *Jatropha curcas* seed oil, Deng *et al.,* (2011) investigated the effect of nanosized solid base catalyst on yields during production of biodiesel from *Jatropha curcas* oil. A biodiesel yield of 95.2% yield was achieved at an optimum condition of alcohol-to-oil molar ratio of 4:1, 1wt% catalyst, 318K at 1.5h reaction time. The produced biodiesel was reported to have similar properties with that of German standard, and the calcined catalyst can be reused for up to 8times. However, the use of nanoparticles catalyst could be cost effective on commercial scale biodiesel production. The aforementioned reviews of the literature shows that despite Nigeria interest into commercial production of biodiesel from *Jatropha curcas* seed, there are still limited studies available on biodiesel production from *Jatropha curcas*. Fundamental information such as mass and energy balance, cost analysis involved in producing biodiesel from J*atropha curcas* are unavailable in the scientific literature. Furthermore, kinetic analysis are important to have data on the design of reaction systems and to promote industrial application of transesterification technology. However, there are strong disagreement on the order of reaction during transesterification of oils to biodiesel. As some investigations have either reported a first order reaction or second order reaction. Thus a good understanding of the chemistry involved in the transesterification of oils to biodiesel is necessary. In addition, products yields are inconsistent despite adhering to similar operating conditions. Moreover, there are several reports on the extraction and transesterification of resultant oil to biodiesel, however there are limited data on the amount of energy involved in the production process. Hence studies on the amount of employed energy and mass balances for a unit biodiesel production are necessary, in order improve understanding of biodiesel production from *Jatropha curcas* seed.Therefore, the aim of this work is to investigate the production of biodiesel from *Jatropha* oil under different reaction conditions. This aim will be achieved with the following objectives: to produce and characterize biodiesel, to elucidate the material and energy balance of the process.

MATERIAL AND METHODS

Materials: Jatropha curcas seeds were obtained from Ozoro in Isoko North L.G.A, Delta State, Nigeria. All the chemicals such as potassium hydroxide, sodium hydroxide, sulphuric acid, methanol, n-Hexane, and ethanol used in this study were analytical grade

Extraction of Jatropha oil: The *Jatropha curcas* seeds were cracked, and the shell removed to obtain the seed kernels, which were sundried to about 5% moisture content. Then the dried seed kernels were grinded prior to oil extraction using a soxhlet extractor using n-hexane as solvent. The soxhlet experimental set-up was heated with a heating mantle to about 80^{o}C, to enable relux of solvent. After which mixture of Jatropha oil and n-Hexane were obtained as product. Then n-Hexane was evaporated from the mixture, leaving Jatropha oil. The resultant oil was analysed for its physiochemical properties prior to biodiesel production. The free fatty acid value of the resultant Jatropha oil was reduced according to the method explained by Aranisola *et al.,* (2012) prior biodiesel production. Free fatty acid not more that 1% is recommended for high yields of biodiesel. The mass balance and energy input of the extraction process were estimated.

Transesterification of Jatropha curcas oil: The transesterification experiment was carried out in batch mode at a reaction temperature of 333K using sodium hydroxide catalyst concentration of 1w/w to *Jatropha curcas* oil at various methanol to Jatropha oil ratio of 1:1, 2:1, 3:1, 4:1, 5:1 and 6:1, a reaction time of 30, 60, 90, 120 and 180min. The experimental procedure was conducted according methods explained by previous research investigations Aranisola *et al*., (2012); Deng *et al*., (2011); Noureddini and Zhu, 1997). Briefly, a sample weight of Jatropha oil and methanol was heated to predefined reaction temperature at varied reaction time. The reaction time commenced after attaining the reaction temperature. Then the catalyst was added, followed by stirring at a constant 200rpm. After complete reaction, the product mixture was allowed to cool to room temperature. Then the product mixture was transferred to a separating funnel and allowed to sit for about 8hr for gravity separation. After cooling, a golden like solution now referred to as biodiesel mixture was formed at the top of the separating funnel, while a light brown solution referred to as glycerol was at the

bottom. Then the glycerol was drained off leaving behind the biodiesel mixture. The biodiesel solution was washed with distilled water up to times, in order to remove traces of unreacted NaOH catalyst and glycerol present in the biodiesel. After washing, the biodiesel solution was subjected to rotatory evaporation to remove residual methanol, and dried in a desiccator. The final solution now referred to as biodiesel was analysed for its physiochemical properties.

Analysis: The ash content, protein, crude fibre and moisture content of the *Jatropha curcas* were determined according the methods explained by AOAC, (2000). The oil yield from *Jatropha curcas* was determined using Eq. (1).

$$Oil\ yield\ (wt\%) = \frac{Mass\ of\ Jatropha\ oil}{Mass\ of\ Jatropha\ curcas\ seed} * 100\% \quad (1)$$

The combined total amount of heat input to produce a unit biodiesel from *Jatropha curcas* seed by heating from room temperature (25^{o}C) to predefined temperature with no vaporization (and assuming heat loss to the environment negligible) (Eboibi *et al*., 2015) was estimated (using Eq. 2) using the enthalpies of saturated liquids.

$$E_{in} = \frac{((M_{n\text{-}H} \times C_{pn\text{-}H} \times \Delta T) + (M_{n\text{-}H} \times HVAP_{n\text{-}H} \times \Delta T) + (M_{me} \times C_{Pme} \times \Delta T) + (M_{me} \times HVAP_{me} \times \Delta T))}{M_{mf}} \quad (2)$$

where $M_{n\text{-}H,}$ M_{me},and M_{mf}, is the mass of n-Hexane, methanol and feedstock, respectively, $C_{pn\text{-}H}$, the heat capacity of n-Hexane (2.76KJ/kgK), and C_{Pme} is the heat capacity of methanol (0.081KJ/kgK), $HVAP_{n\text{-}H}$ is the latent heat of vaporization of n-Hexane (28.85KJ/mol), $HVAP_{me}$ the latent heat of vaporization of methanol (37.43KJ/mol), ΔT is the temperature change. The heat input for temperature rise was estimated using $M_{n\text{-}H}\, C_{pn\text{-}H}\, \Delta T$ and $M_{me}\, C_{Pme}\, \Delta T$ for n-Hexane and methanol respectively, while $M_{me} C_{Pme}\, \Delta T$ were used determine the heat of vaporization of n-Hexane and M_{me} $HVAP_{me} \Delta T$ for vaporization of methanol. It should be noted that the energy required for grinding the *Jatropha curcas* seed and the time for heating was not considered in the Eq. (2).

The physiochemical properties of the extracted oil and the produced biodiesel were analysed according to the methods explained by AOAC, (2000) and Aranisola, (2010). The flash point for the produced biodiesel was determined using a method normally referred to as flash cup closed tester. Here a sample of biodiesel is placed in a coning flask, then mounted on a heater attached with a thermometer and closed tightly. Then the flask is slowly heated and thoroughly stirred. Small flames were then directed into the cup at regular intervals of 2min with simultaneous interruption of stirring. The temperature at which the sample flashed (i.e. the temperature at which the vapour of the biodiesel ignited and turned off again after the ignition source was removed) was recorded as the flash point. Thereafter, the flask was placed in a cooling bath and was examined for its cloud point. The temperature at which the biodiesel sample was cloudy was observed and recorded as the cloud point. The viscosity and density was determined using Ostwald viscometer, and the density by standard method.

RESULTS AND DISCUSSION

Biochemical composition of Jatropha curcas: The biochemical composition of *Jatropha curcas* seed is presented in Table 1. The ash content were 3.5w/w%, 24w/w% for protein, 20w/w% for crude fibre and 5w/w% for the moisture content. It was found that the obtained data were within the range of previous research investigations (Aranisola *et al*., 2012, Knutson *et al*., 1983), as shown in Table 1.

Table 1: Biochemical composition of *Jatropha curcas* seed

Properties	**Present study**	**Aranisola *et al*., (2012)**	**Knutson *et al*., (1983)**
Ash	3.5	3.25	6.2
Protein	24.0	23.2	21.0
Crude fibre	20.0	23.12	19.92
Moisture content	5.0	4.03	4.05

*Biodiesel yield***:** This section discusses the yield of biodiesel obtained at different methanol-to-oil ratio and reaction times following transesterification of Jatropha oil. Generally, increase in methanol-to-oil led to an increase in biodiesel yield. Biodiesel yield obtained at methanol-to-oil ratio 1:1 at different reaction times is presented in Figure 1. As shown in Figure 1, biodiesel yield increase with an increase in reaction time, as mentioned previously. Maximum biodiesel yield of 45wt% was achieved at 180min, minimum 28wt% at 0.5hr (30min).

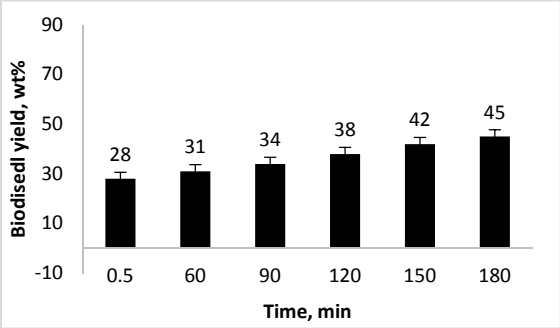

Fig 1: Biodiesel yield at methanol-to-oil ratio 1:1 at different reaction times

For methanol-to-oil ratio 2:1, the yield of biodiesel is shown in Figure 2. As illustrated in Figure 2, 35wt% biodiesel yield was achieved at 0.5hr compared 28wt% obtained at methanol-to-oil ratio 1:1. Biodiesel yield increased from 35wt% to 37wt% at 60min, and with maximum yield of 48wt% at 180min. This increase could be due to increase concentration of methyl ester.

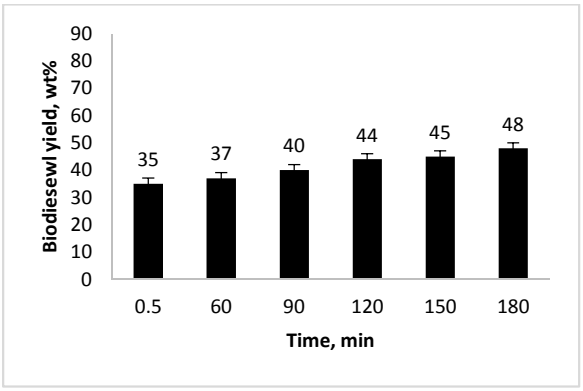

Fig 2: Biodiesel yield at methanol-to-oil ratio 2:1 at different reaction times**.**

Similarly, there were increase yields of biodiesel with an increase in reaction times at methanol-to-oil ratio 3:1 (shown in Figure 3). As shown in Figure 3, the increase in biodiesel yields were higher when compared to methanol-to-oil ratio of 2:1. The variation in yields were between 3wt% and 6wt% compared to 2wt% and 3wt% for methanol-to-oil ratio 2:1. Maximum yield of 63wt% were obtained at 180min reaction time, while 39wt% minimum was achieved at 0.5hr. Moreover, biodiesel yields at ratio 3:1 were found to be generally higher than yields obtained at ratios 2:1 and 1:1.

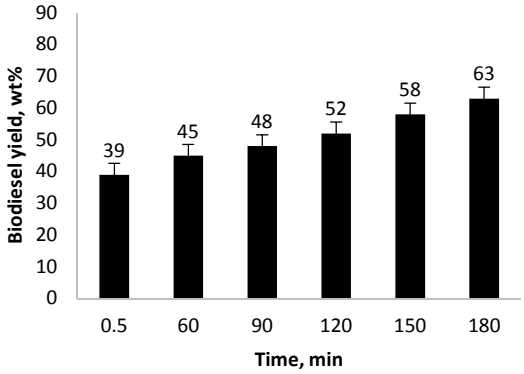

Fig 3: Biodiesel yield at methanol-to-oil ratio 3:1 at different reaction times.

Biodiesel yields obtained at methanol-to-oil ratio 4:1 is presented in Figure 4. Based on the data shown in Figure 4, there were increase in biodiesel yields with an increase in reaction times. At 0.5h reaction time 42wt% biodiesel yield were derived, and further increase of reaction time led to continua increase of biodiesel yield from 42wt% to 68wt at 180min. The variation in yield were higher at lower reaction time between 0.5hr and 60min compared to above 60min. Similar trend was observed for lower methanol-to-oil ratios except ratio 1:1.

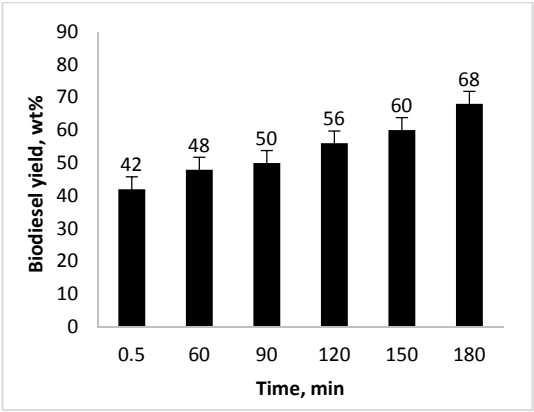

Fig 4: Biodiesel yield at methanol-to-oil ratio 4:1 at different reaction times.

The data obtained following transesterification of *Jatropha curcas* oil at methanol-to-oil ratio 5:1 is presented in Figure 5. The biodiesel yield follows similar trend in terms of increase in from 45wt% to 72wt% at 0.5hr to 180min. maximum yield of 72wt% were obtained at 180min while 45wt% for minimum. Compared to previous ratios, the biodiesel yields obtained at different reaction times were generally

higher, suggesting that higher methanol-to-oil ratios could still lead to increase in yields.

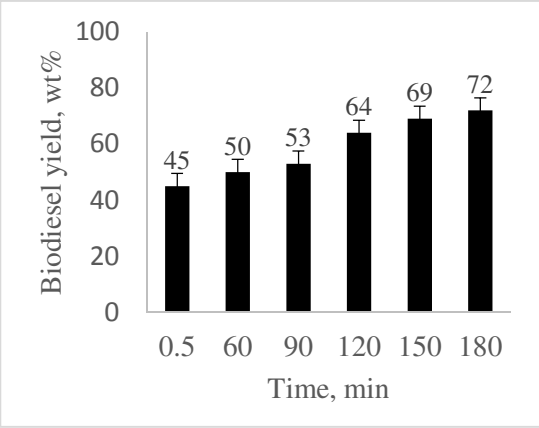

Fig 5: Biodiesel yield at methanol-to-oil ratio 5:1 at different reaction times**.**

For methanol-to-oil ratio 6:1, the biodiesel yields achieved at 0.5hr to 180min reaction times are presented in Figure 6. As mentioned previously, there were general increase in yields of biodiesel with an increase in reaction times. Biodiesel yield increase from 50wt% to 86wt% at 0.5hr to 3hr (180min). In contrast to previous ratios, longer reaction times favoured higher incremental yields. Yields obtained at different reaction times were found to be generally higher compared to those achieved at ratios 1:1 to 5:1.

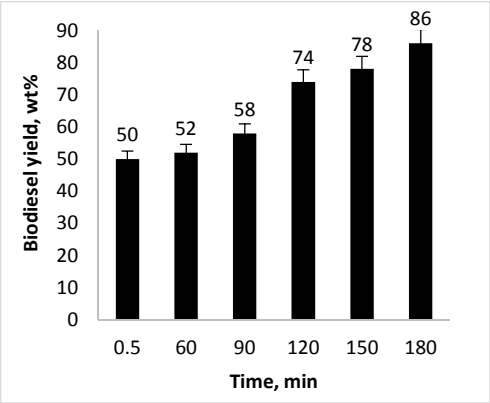

Fig 6: Biodiesel yield at methanol-to-oil ratio 6:1 at different reaction times.

In summary, the present study has shown that biodiesel could be produced from *Jatropha curcas* seed. Longer reaction times led to an increase in product yields, which could be due to methanol content being above stoichiometric value, hence the enhanced formation of reaction products. At methanol-to-Jatropha oil ratio of 1:1, the maximum yield in biodiesel were 45wt% at 180min, while it was 66wt%, 76wt%, 77wt%, and 86wt% at ratio 2:1, 3:1, 4:1, 5:1 and 6:1, respectively, at similar reaction time. Based on the data presented in Figure 1 to Figure 6, an increase in methanol-to-oil ratio led to an increase in concentration of methyl ester.

In addition, the reported yields were found to be within the range of previous investigations (Aranisloa *et al*., 2012; Wang *et al*., 2011). Aranisola *et al*., (2012) reported 87wt% at ratio 6:1 and 60^{o}C. Wang *et al*., (2011) reported a maximum yield of 86.2% biodiesel following alkali transesterification of *Jatropha curcas* oil at methanol-to-oil ratio of 6:1, 1% w/w alkali catalyst and at a reaction temperature of 60^{o}C. Moreover, this present study has shown that that further increase in reaction time and molar ratio would enhance the recovery of biodiesel. Freedman *et al*., (1986) reported that about 80% of conversion occurred within few minutes of reaction, then after 60min the yield in ester conversion increased to a range of 93wt% to 97wt%. Although, Freedman *et al*., (1986); Sarve *et al*., (2015) and Deng *et al*., (2011) reported higher yields (94.7wt% to 98.14%) compared to those obtained in the present study, it could be due temperature of transesterification, catalyst and the quantity of employed alcohol.

Importantly, the trend in biodiesel yield with increase in methanol-to-oil ratio were found to be consistent with previous research investigations, suggesting that molar ratio of methanol-to-oil has significant effect on biodiesel yield. This is agreement with previous research investigating biodiesel production from *Jatropha curcas* seed (Aranisola *et al*., 2012; Jain and Sharma, 2010; Wang *et al*., 2011). For example, Aranisola, *et al.*, (2012) reported that an increase in the molar ratio from 3:1 to 6:1 led to higher yields of biodiesel, where a maximum yield (87wt%) of biodiesel was obtained at a ratio of 6:1 at 333k.

Furthermore, the physiochemical properties of the Jatropha oil and produced biodiesel are shown in Table 2. Based on the data presented in Table 2, the values obtained from the analysis of the Jatropha biodiesel especially flash point, water content and viscosity of the oil were found to be within the range of the standard biodiesel. Suggesting that the extracted Jatropha oil was suitable for biodiesel production. In addition, the saponification value of the extracted *Jatropha* oil being 190 mg KOH/g, suggests that the oil could be useful for soap making. This high value (190 mg KOH/g) was in accordance with the previous report shown in the Table 2. Saponification value is an important parameter in Jatropha oil production as

higher saponification values indicate the normality of the oil as triglyceride which is very useful in production of liquid soap and shampoo. Saponification value also renders the separation of ester and glycerol difficult since it increases the viscosity and form gels. Moreover, the acid value of *Jatropha* oil was 8mgKOH/g, which implies low fatty acid content compare to findings in literature. The low in acid value might be as a result of some factors which include the type of feedstock used for Jatropha oil production, production process and its respective degree of purification. The acid value gives an indication of the quality of fatty acids in the oil. The free fatty acid of the extracted Jatropha oil was 4%, however, high free fatty acid content is unfavourable in an alkali-catalysed transesterification reaction because the free fatty acid will react with the catalyst to form soap and separation of products will be extremely difficult, resulting in a low yield in biodiesel. For free fatty acid >1%, acid catalysed transesterification can be used directly without reducing the free fatty acid. Though the acid catalysed transesterification is slower compared to alkali catalysed. Because of the slow in acid catalysed transesterification, alkali-catalysed transesterification was used in this study in accordance to Aranisola *et al*., (2012).

Table 2: *Physiochemical properties of oil obtained Jatropha curcas seed.*

Property	**Jatropha oil (present study)**	**Jatropha Biodiesel (present study)**	**Aranisola *et al*., (2012)**	**Biodiesel standard**
Acid value	8	-		<0.80
Cloud point (^oC)	11	8	3	-
Density (15°C, kgm^3)	895	870		
Flash point (^oC)	-	146	170	>130
Free fatty acid	4	-	-	-
Pour point (^oC)	5	2	-6	-
Saponification value (mgKOH/g)	190	-	-	-
Specific gravity	0.91	0.92	0.88	0.860-0.900
Viscosity (mm^2s-^{1})	2.98	2.65	5.64	1.9-6.0
Refractive index	1.4558	1.4611		
Moisture content	0.2	0.05	0.0	<0.03

Table 3: Mass balance during production of biodiesel from *Jatropha curcas* seed.

Extractor Unit		**Transesterification Unit**	
Input	**Output, wt%**	**Input, wt%**	**Output**
Grinded *Jatropha curcas* seed, 1kg n-Hexane l	Jatropha oil ,42.5 Waste (seed cake), 51.5 Loss, 6 n-Hexane	Jatropha oil, 42.5	Biodiesel, 28 to 86

Table 4: Energy analysis during production of biodiesel

Feedstock	**Production process**	**E_{in} MJ/kg**	**E_{out}= $HHV_{biodiesel}$ MJ/kg**	**$\Delta E = E_{out} - E_{in}$ MJ/kg $_{biodiesel}$**	**Reference**
Jatropha curcas seed	Extraction and transesterification	1.4	38.5*	37.1	Present study
Jatropha curcas	Extraction and transesterification	NR	NR		Deng *et al*., (2011)
Jatropha seeds	Extraction and transesterification	NR	NR		Kadry, (2015)
Jatropha and castor oils	Transesterification	NR	38.5		Thanachayan *et al*., (2013)*
Castor oil	Transesterification	NR	NR		Oladimeji and Oyekunle, (2015)
Soybean oil	Transesterification	NR	NR		Nourddini and Zhu, (1997)
Vegetable oil	Transesterification	NR	NR		Sanjel *et al*., (2014)
Schleichera triguga	Extraction and transesterification	NR	NR		Sarve *et al*., (2015)
Soybean oil	Transesterification	NR	NR		Freedman *et al*., (1986)
Mineral biodiesel	Conventional process		45.34		Thanachayan *et al*., (2013)

E_{in}: heat input, E_{out}: heat output, ND: not determined, NR: not reported; HHV = Higher heating value

For alkali-catalysed transesterification, it has been reported by researchers that transesterification will not occur if the free fatty acid is above 1% which is why the Jatropha oil free fatty acid content were reduced.

Viscosity is the measure of material resistance to flow, higher viscosity materials flows with great difficulty and a material with less viscosity flow more easily. Viscosity is important to diesels and biodiesels because it has impacts on the operation of some engine components such as the fuel pump. The viscosity of the biodiesel produced (2.65mm^2/s) is lower than that reported by Aranisola *et al*., (2012), however it is within the specified range of the ASTM standard (1.9mm^2/s to 6.0mm^2/s). Other parameters tested were within the limits of ASTM confirming that biodiesel produced from the study met the criteria for acceptable standards.

*Mass and energy balance***:** The extraction of the oil from the Jatropha oil seed was performed in batches. The average yield of the primary product Jatropha oil, cake and losses are presented in Table 3. As shown in Table 3, the *Jatropha curcas* seed has 42.5w/w% oil content, 51.5w/w% cake and about 6w/w% was observed. Other studies have reported similar findings, however it could be possible to have higher yields with increase in time. The oil yield obtained in the present study was found to be within the range of previous reports. For example, Kadry, (2015) reported 41.30% to 45%, an oil yield of 47.5% and 49.1% was reported by Akintayo, (2004) and Martin *et al*., (2010), respectively. Interestingly the oil content oil Jatropha seed was found higher when compare to those of soybean (18.35%), linseed (33.3%), suggesting its potential as a non-edible vegetable oil feedstock for biodiesel production. As mentioned previously, there are limited data on the amount of energy input during the production of biodiesel from *Jatropha curcas.* Hence the reported data would provide additional knowledge. For the process to be more economical, the product seed cake could be further processed.

The heat input required for producing of a unit biodiesel is presented in Table 4. As shown in Table 4, the amount of energy required (excluding the energy for grinding and reaction time) was found to be 1.4MJ/kg. To the best of our knowledge, this is the first report to have estimated the energy input for the production of biodiesel from *Jatropha curcas* seed. The production process seems to have a net energy production, since the energy output was 38.5MJ/kg (Thanachayan *et al*., 2013). Although consideration of both the reaction time and the energy used in grinding would increase the total combined energy input in the process, there is still the feasibility to yield a net energy production. Nevertheless, the entire production process needs improvement especially in terms of product yields, reduction in waste products, reaction time and feedstocks. Also research into the energy conversion ratio would enhance an understanding of the process, as Table 4 clearly shows that there are limited report on the energy involved during production of diesel.

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