
Assessment of Milk Bush Seed Oil as an Auspicious Feedstock for Biodiesel Fuel

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Abstract: It is indubitable that world energy demand is increasing drastically due to rapidly growing population and urbanization. It is not obscure that biodiesel would make a massively copious contribution to the energy demand at a time when the populace is becoming increasingly conscious of the declining reserves of fossil fuels and detrimental environmental effects it poses. There are several potential feedstocks that can be used for biodiesel production. The second generation feedstocks which are the non-edible vegetable oils could be considered as promising replacement for first generation feedstocks which are the edible vegetable oils. The usage of non-edible vegetable oil in the production of biodiesel is very significant because of the profuse demand for edible oils as food source. Moreover, first generation's feedstock costs are exorbitant to be used as fuel. However, in this study, non-edible milk bush (*Thevetia peruviana*) seed in which its seed is enrich with oil and can be grown in arid and semi-arid condition, on waste land, roadsides and road-dividers in expressways for beautification, environmental protection and hated by herbivorous animals, was used in biodiesel production. The oil was extracted with n-hexane using soxhlet apparatus with which ample amount (60.2%) of oil was extracted. Biodiesel was produced via trans-esterification process from the crude oil of milk bush (*Thevetia peruviana*) seed with methanol. The optimum condition was obtained at molar ratio 6:1 of alcohol to oil, temperature of 55°C, reaction time of 60 minutes and sodium hydroxide as the base catalyst adopted. Some fuel properties (kinematic viscosity, centane number, flash point, density, cloud point, acid value and moisture) of the biodiesel produced were determined. Results obtained for these fuel properties are 4.48 mm²/s, 55, 135°C, 0.866, 4, 0.1 mgKOH/g, 0.03% for kinematic viscosity at 40°C, centane number, flash point, density, cloud point, acid value and moisture content respectively. The results obtained were in agreement with ASTM D6751 and EN 14214 standards. In conclusion, it has been found that there is an immense chance to produce biodiesel from milk bush (*Thevetia peruviana*) seed oil and therefore it can boost the future production of biodiesel.

Keywords: Biodiesel, Milkbush, Non-edible Oil, Trans-esterification, *Thevetia Peruviana*

1. Introduction

The global increasing environmental and energy concerns caused by declining petroleum reserves and emission of green-house gases from fossil fuels have made biodiesel fuel a promising attractive energy source for the future. Biodiesel is non-petroleum based alternative source of energy. It can be defined as long chain fatty acids of mono-alkyl esters obtained from vegetable oils or animal fats and alcohol with or without a catalyst [1]. It has gained and revived the world's interest because of its sustainability, renewability, non-toxicity, availability, economic and environmental friendly advantage [2].

Vegetable oils and animal fats are the major feedstock for the production of biodiesel. The vegetable oil could be edible and non-edible but the apparent ever increasing demand of edible oils for food renders the use of non-edible oil attractive for biodiesel production [3]. Some examples of non-edible seed oil crops are; *Madhuca indica* (mahua), *Jatropha curcas*, *M. azedarach* (syringe), *Pongamia pinnata* (karanja), *Hevea brasiliensis* (Rubber seed), *Azadirachta indica* (neem), *Simmondsia chinensis* (Jojoba), *Putranjiva roxburghii* (Lucky bean tree), *Sapindus mukorossi* (Soapnut), *Thevetia peruviana* (yellow oleander/milk bush), etc. For the purpose of this study, milk bush seed oil would be adopted for the production of biodiesel.

Milk bush (*Thevetia peruviana*), more commonly known as yellow oleander belongs to the family Apocyanaceae which is an evergreen ornamental dicotyledonous shrub that can be found in the tropics and sub tropics region of the world [4]. It is abundantly grown in Nigeria for ornamental purpose since it adapts so well to arid and semi-arid condition and also requires low fertility and moisture to grow [5]. The plant's seed contains about 60% oil, which hydrolyses to give about 64.3% oleic acid, 6.3% linoleic, 17.1% palmitic, 11.8% stearic, 0.4% arachidonic acid and also the cake comprise of 30 – 37% protein [6]. Due to the high oil value of the seed, it has a great potential to be used as a feedstock for biodiesel. Aside the high oil content of milk bush seed, it has numerous advantages which improves engine performance which are the higher heat content, lower sulphur content, lower aromatic content and the seed cakes after oil extraction can serve as fertilizers which enriches soil [7]. All parts of the plant are toxic due to the presence of cardiac glycoside which makes it non edible [8]. The non-edibility renders it more auspicious since it doesn't compete for use as food, effort should be massively intensified and explored for its maximum use in the production of biodiesel.

Conventionally, the synthesis of biodiesel from vegetable oils involves isolation of the oil from the seed and then transesterification with alcohol in the presence of an acid or base catalyst. It has been shown through various researches that the oil can be extracted using soxhlet extraction or mechanical pressing method. Moreover, the conversion of the triglyceride into an alkyl ester could be achieved through any of the following methods; blending, thermal cracking (pyrolysis), trans-esterification and micro-emulsion [9]. Trans-esterification of triglyceride with monohydric alcohol is the most commonly adopted method for the production of biodiesel from animal fats or vegetable oils with or without catalyst and has been studied for several decades and a large part has been achieved industrially using this method [10]. Therefore, the objective of this study was thus to investigate the detail process of base catalyzed trans-esterification of

crude milk bush seed oil with methanol. Alongside, the influence of methanol to seed oil ratio, amount of alkali (KOH) catalyst, temperature and reaction time was examined to identify the optimal reaction conditions and define best performance of biodiesel yield and quality. The fuel properties of milk bush seed oil methyl-esters (biodiesel) in comparison with the accepted biodiesel standards were also investigated.

2. Materials and Method

2.1. Materials

Fresh matured fruits of milk bush (*T. peruviana*) were obtained from Ehirobo street, Bells University drive, Sango Ota, Ogun State, Nigeria on the 4th of March, 2016. The fruits were immediately transported to the laboratory where they were manually separated to remove all forms of impurities. The clean seeds were dried at room temperature for several days after which they were milled with blender. Methanol (> 98% purity), n-hexane (> 98% purity) and all solvents and chemicals used were pure and of analytical grades.

2.2. Oil Extraction

A known weight of the dried milled milk bush seed was subjected to soxhlet extraction using two hundred millilitres (200 ml) of n-hexane which was placed in a 500 ml round bottom flask of the soxhlet apparatus. The Soxhlet apparatus was mounted to a heating mantle and was heated between 60°C - 65°C so as to prevent loss of hexane vapour because n-hexane has a boiling point of 69°C. The evaporating n-hexane was condensed into the thimble by the condenser where it leached the oil out of the paste. The oil rich solvent after reaching a level siphoned back automatically into the round bottom flask where the process described so far was repeated. The solvent was recovered using rotary evaporator which leaves behind solvent free milk bush seed oil and it was weighed to calculate the percentage yield [11].



Figure 1. Milk bush plant with its fruit and seed.

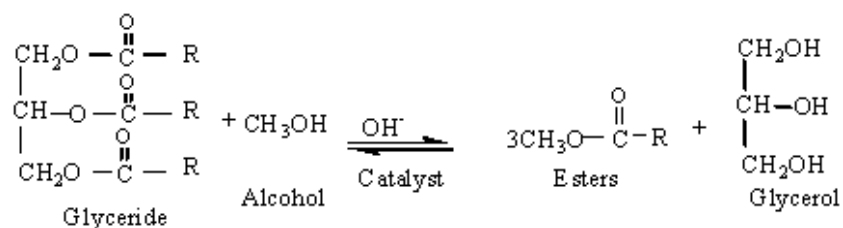


Figure 2. Chemical equation of the trans-esterification of triglyceride with methanol using base catalyst (Singh, 2010) [11].

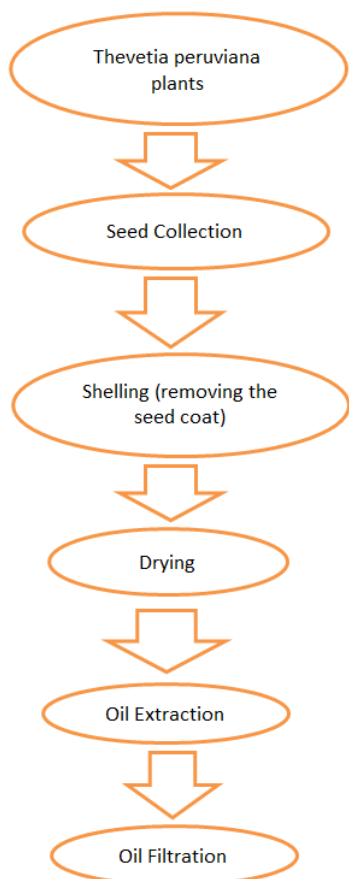


Figure 3. Sample preparation flowchart of *Thevetia peruviana* seed oil [11].

2.3. Analysis of *Thevetia Peruviana* Oil

Some physico-chemical properties with which vegetable oils are identified as recommended by American Standard Testing Method (ASTM), Association of Official Analytical Chemists (AOAC) and other standards were determined for the extracted milk bush (*Thevetia peruviana*) seed oil. These are specific gravity, refractive index, percentage free fatty acid (FFA), saponification value, viscosity, peroxide value and moisture content.

2.4. Preparation of Catalysts

The amount and type of catalyst adopted is one of the important factors that determine the yield of biodiesel. The essence of the titration process is to get the number of gram of NaOH/KOH that would be used in the trans-esterification process. A lye was prepared using sodium hydroxide (NaOH) in which 1 gram of NaOH was dissolved in 1 Litre (L) of distilled water to make 0.1% lye. 10mL of isopropyl alcohol was used to dissolve 1g of crude milk bush seed oil which was warmed gently in a water bath until it completely dissolved. Thereafter, 2 drops of phenolphthaleine solution was added to the mixture in which 0.1% NaOH was titrated against the mixture until a stable pink colour was formed. The average titre value of NaOH in addition to 4g of NaOH (catalyst) will be needed for transesterification of the milk bush seed oil extracted [12].

2.5. Production of Biodiesel (Transesterification of Milk Bush Seed Oil)

The crude milk bush seed oil was trans-esterified in batches to calculate the yield of biodiesel. 250mL of milk bush seed oil was measured into a three necked flask fitted to a thermometer and liebig condenser and was heated to 50°C in a closed system. Thereafter, 3.75g of NaOH was dissolved in 50mL of methanol in a covered bottle and shaken thoroughly to dissolve the salt. The methanol and sodium hydroxide mixture (methoxide) was poured into the heated oil and simultaneously heated and stirred using a magnetic stirrer bar at a temperature of 55°C.

The mixture was allowed to cool, transferred to the separating funnel and allowed to stand for 24 hours for good separation. After 24 hours, two distinct layers were observed, in which the upper layer is the fatty acid methyl ester (biodiesel) and the lower layer is the glycerol because glycerol is heavier than the biodiesel formed. The biodiesel was washed with warm distilled water for four to five times until a clear solution was observed in the lower layer, after the completion of washing process the biodiesel may contain some traces of water. Biodiesel was placed on a water bath heated to 105°C to remove the trapped traces of water (for drying) [12].

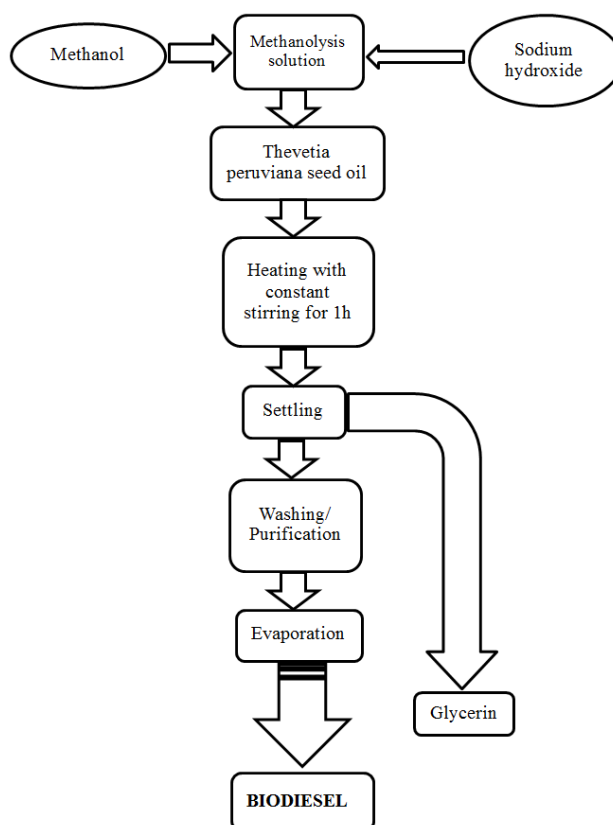


Figure 4. Trans-esterification flow process for biodiesel production [13].

3. Results and Discussion

After extraction, oil extracted from milkbush seed was

golden yellow in colour and free of sediments. The percentage of oil from the seed of *Thevetia peruviana* was determined. The homogeneous base catalysed trans-esterification of the extracted oil and physico-chemical properties of oil and biodiesel were carried out. In addition, the optimum conditions such as oil/methanol molar ratio, reaction temperature and time, effect of catalyst were investigated to determine highest yield of the biodiesel quantitatively.

3.1. Percentage Oil Yield from Milk Bush Seed

Golden yellow oil was obtained from the milk bush seed after extraction. The percentage of oil extracted is shown in Table 1.

Table 1. Percentage of Oil Extracted.

W ₁ (g)	W ₂ (g)	% Oil yield
1000	398	60.2

W₁ is the weight of the sample before extraction. W₂ is the weight of the sample after extraction.

Table 3. Comparison of the fuel properties of the biodiesel produced from milk bush seed oil with American Society of Testing and Materials (ASTM) biodiesel standard [16].

Property	Present Study	ASTM D6751 Biodiesel standard	Test method	EN 14214 Biodiesel Standard
Flash point (°C)	135	130.0 min	ASTM D93	120 min
Moisture content (%)	0.03	0.050 max	ASTM D2709	0.05 max
Density	0.866	0.86-0.900	ASTM D1928	
Kinematic Viscosity at 40 °C (mm ² /s)	4.48	1.9 – 6.0	ASTM D445	3.5 – 5.0
Centane Number	55	47 min	ASTM D613	51 min
Cloud point (°C)	4	3 to 12	ASTM 2500
Acid value (mgKOH/g)	0.1	0.5 max	ASTM D664	0.5 max

Table 4. Fuel properties of *T. peruviana* methyl ester (TPME) reported in various literatures and their comparison with the present study.

Properties	Present study	Adebowale et al. [17]	Deka et al. [18]	Betiku et al. [19]
Flash point (°C)	135	125	75	196
Cloud point (°C)	12	----	12	8
Density	0.866	0.870	0.875	0.887
Kinematic Viscosity at 40°C (mm ² /s)	4.48	4.50	4.33	6.0
Centane Number	55.2	54.2	61.5	123.5
Acid Value (mg KOH/g)	0.100	0.20	0.057	0.46
Moisture content (%)	0.03	----	----	----

3.2. Fuel Properties of Biodiesel Produced from *Thevetia Peruviana* Seed Oil

The properties such as the kinematic viscosity at 40°C, density at 25°C, flash point, cloud point, acid value, moisture content, centane number were investigated and reported.

3.2.1. Kinematic Viscosity

The kinematic viscosity at 40°C of TPME was determined to be 4.48 mm²/s which is within the biodiesel standard set by ASTM D6751 and EN 14214 with prescribe viscosity ranges of 1.9–6.0 and 3.5–5.0 mm²/s, respectively. The value (4.48 mm²/s) agrees well with 4.50 mm²/s and 4.33 mm²/s reported by Adebowale et al. [17] and Deka et al. [18] respectively, who produced methyl esters (biodiesel) from the oil of *Thevetia peruviana*. Also, Betiku et al. [19] produced biodiesel from yellow oleander oil with kinematic viscosity

This value showed high oil yields. Milk bush seed oil has been worked on extensively and the percentage oil yields have been found to be promising and comparable with the values obtained in other studies. Ibiyemi, 2002 [14] found appreciable percentage oil yields of 64% for *Y. Oleander*. Olisakwe et al. 2011 [15] also reported a little higher yield for *Jatropha* and *Yellow oleander* (54.6% and 58.5% respectively).

Table 2. Physico-chemical parameters of the extracted oil.

S/N	PARAMETERS	VALUES
1	Refractive index	1.4660
2	Specific gravity	0.912
3	Acid value	4.2
4	Free Fatty Acid	2.1
5	Saponification value	60.68
6	Peroxide value	3.9
7	Moisture content (%)	0.58
8	Viscosity at 40°C (Pa/s)	7.8

value of 6.0 mm²/s which is slightly higher than the present study (4.48 mm²/s) and falls above 3.5–5.0 mm²/s biodiesel standard set by ASTM D6751 but falls within that of EN 14214.

3.2.2. Cetane Number

For the determination of diesel fuel quality, especially the ignition quality, the cetane number is an important parameter which measures the readiness of the fuel to auto-ignite when it is injected into the engine [20]. As shown in Table 3, the cetane number of TPME was determined to be 55.2 which is in agreement with 54.2 reported by Adebowale et al. [17] and considerably lower compared to 61.5 reported by Deka et al. [18]. High centane number 123.5 reported by Betiku et al. [19] implies short ignition delay and fuels with low centane number tend to cause

incomplete combustion as a result of increased gaseous and particulate exhaust emissions [21]. The cetane number of *Thevetia peruviana* derived biodiesel investigated in this study agrees with the minimum cetane number requirements in both the ASTM D6751 and EN 14214 biodiesel standards, which are 47 and 51, respectively.

3.2.3. Flash Point

Flash point is another important property of biodiesel fuel, which is the lowest temperature at which it will spontaneously ignite when exposed to a flame or spark. Biodiesels have quite high flash points which make them less volatile and safer to transport or handle compare to diesel fuel. As shown in Table 3, the flash point of TPME fuel is 135°C which can be compared well with ASTM D6751 and EN 14214 biodiesel specifications with the minimum of 130°C and 120°C respectively. It can also be compared with that reported by Adebowale et al. [17] who produced methyl esters (biodiesel) from the oil of *Thevetia peruviana* and the flash point was found to be 125°C which is slightly below 130°C set by ASTM D6751 and slightly above 120°C set by EN 14214 biodiesel specifications. Deka et al. [18] also produced biodiesel from the oil of *Thevetia peruviana* with flash point 75°C that is drastically below the biodiesel specifications.

3.2.4. Density

Density is the relationship between the mass and volume of any liquid or a solid at a given temperature expressed in units of grams per liter (g/L). The density of diesel oil gives an indication of the delay between the injection and combustion of the fuel in a diesel engine and the energy per unit mass [22]. The test method ASTM D1298 was adopted to measure the density of the biodiesel. From Table 3, it has been found that TPME has density of 0.867 g/cm³ which is in conformity with the standard 0.86 – 0.9 g/cm³ set by ASTM D6751. According to these standards, density should be tested at the temperature reference of 15°C [23]. Hotti and Hebbal, 2015 [24] reported that the density of biodiesel obtained from non-edible seed oil is higher than that of conventional diesel fuel and may be attributed to the presence of higher molecular weight triglycerides.

3.2.5. Acid Value

Acid value measures the amount of carboxylic acid groups in a chemical compound, such as a fatty acid, or in a mixture of compounds. Acid value is a relevant parameter which provides indication of the level of lubricant degradation while the fuel is in service [25]. It is usually expressed as mg KOH/g and is set to a maximum value of 0.5 mg KOH/g in the European standard (EN 14104) and ASTM D664. From Table 3, the acid value of TPME was determined to be 0.1 mg KOH/g which falls within the set standard and it is in agreement with the acid values 0.057 mg KOH/g, 0.20 mg KOH/g and 0.46 mg KOH/g reported by deka et al. [18] adebowale et al. [17] and betiku et al. [19] respectively. The presence of higher acid content can cause severe corrosion in internal combustion engine and fuel supply system [25].

3.2.6. Moisture Content

Moisture is a common component that is usually found in all feed-stocks. Moisture reacts with the catalyst during transesterification reaction which leads to soap formation [26]. Biodiesel is generally considered to be insoluble in water and it actually takes up considerably more amount of water than diesel fuel [26]. Hence, before biodiesel production or during ester purification moisture must be eliminated [27]. The moisture content 0.030 wt% in the biodiesel was determined in accordance to EN1412 with specification of 0.050 wt% maximum. However, the moisture content of biodiesel is encouraged to be profusely eliminated to avoid reduction in the heat of combustion which could further cause corrosion of fuel system components [28].

3.3. Effect of Different Reaction Parameters on the Biodiesel Yield

There are several factors that enhance the yield of alkyl ester during transesterification reaction. These variables include alcohol/oil molar ratio, reaction time, reaction temperature, water content, and FFA content [29].

3.3.1. Effect of Molar Ratio of Alcohol to Oil and Alcohol Type

Molar ratio of alcohol to oil is one of the most important factors that affect the yield of alkyl ester. Generally, short chain alcohols such as methanol, ethanol, propanol, and butanol can be used in the trans-esterification reaction while it is encouraged to use the alcohol of shorter chain to obtain high alkyl ester yields [30]. Stoichiometrically, one mol (1 mol) of triglyceride and three mols (3 mol) of alcohol are required in trans-esterification reaction to yield three mols (3 mol) of fatty acid ester and one mol (1 mol) of glycerol [31]. Higher alcohol/oil molar ratios are required to influence the solubility between the triglyceride and alcohol molecules. The alcohol/oil molar ratio of 6:1 or higher generally gives the maximum yield (higher than 98 wt%) [32]. However, the higher molar ratio is required to complete the reaction at higher rate. The yield of methyl ester could be increased by introducing an excess amount of methanol to shift the equilibrium to the right [33]. There was a significant effect on the yield of biodiesel for the variation of methanol to oil molar ratio. The yield of TPME increases as additional methanol was added.

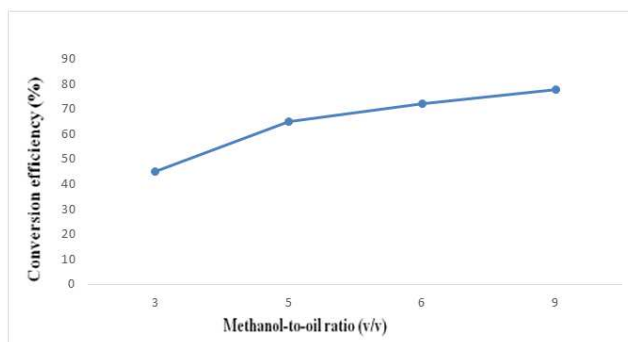


Figure 5. The effect of methanol-to-oil variation on the conversion efficiency.

3.3.2. Effect of Reaction Temperature and Time

The reaction temperature plays an important role in alkaline-catalyst trans-esterification. At room temperature there was no significant yield and it was observed that increase in the reaction temperature had a massive influence on the yield of ester conversion. The effect of temperature variation on conversion efficiency is shown in figure 6. The temperature was varied at four different levels such as 45, 50, 55 and 60°C with which 55°C gave maximum methyl ester yield. There is a greater chance of losing the methanol if the temperature is greater than 60°C, because the melting point of methanol is 63.4°C. Also, with higher temperature, there is possibility of having higher glycerol yields and lower biodiesel yields [34].

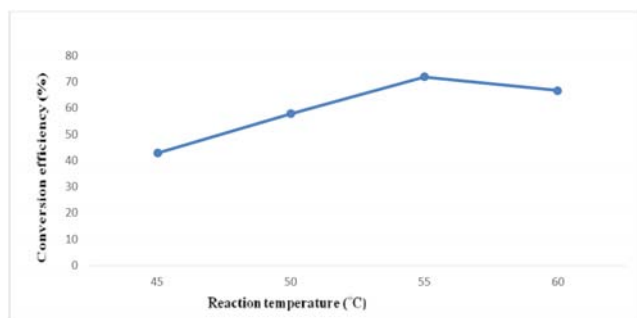


Figure 6. The effect of reaction temperature variation on the conversion efficiency.

Moreover, the methyl ester conversion rate was observed to increase with increase in reaction time. The effect of reaction time variation on the conversion efficiency is shown in figure 7. The maximum efficiency achieved was 72.13% with reaction time of 1 hour.

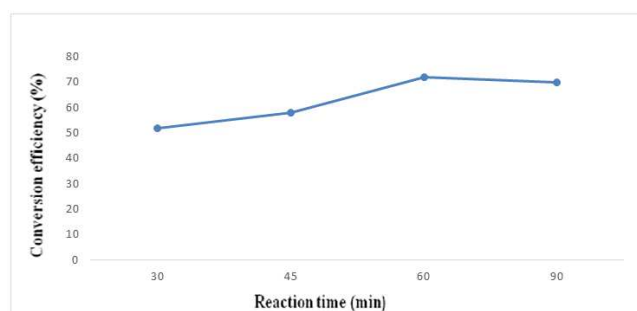


Figure 7. Effect of reaction time variation on the conversion efficiency.

Different reaction time for the transesterification process have been reported by different researchers. Kim et al. (2004) [35] studied the transesterification reaction, with maximum biodiesel yield within 1 hour.

4. Conclusion

Recently, biodiesel which is a mixture of methyl esters of long chain fatty acids has become increasingly attractive because of its apparent economic and environmental benefits. The fuel properties of biodiesel produced from milk bush seed oil determined in this study were found to be

comparable to those of ASTM D6751 and EN 14214 biodiesel standards. Therefore, from this research, it can be concluded that *Thevetia peruviana* seed oil can be a potential alternative to produce biodiesel which could create bio-diversification from over dependency on limited fossil fuel, reduce the threat of global warming and help solve socio-economic problem. Production of biodiesel from low cost non-conventional and non-edible oil resources is most preferred compared to biodiesel from edible oil resource because it can be grown on waste land areas and does not compete with food crops which bring about the issue of food versus fuel.

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