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Effect of Process Parameters on Yield and Conversion of *Jatropha* Biodiesel in a Batch Reactor

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Abstract

In a quest for environmental friendly energy source with least pollutants emission due to issues of global warming coupled with dwindling reserve of the fossil fuel, researchers have intensified study on renewable fuels. Among these renewable energy sources, biodiesel stands prominent. Biodiesel production is largely by transesterification of transglycerides of fatty acids almost always in a batch reactor. Of importance in the yield generation and fatty acid methyl esters conversion is the feedstock purity, control of reagents use in production and operation parameters alteration. This is geared towards achieving optimum resource conservation while also minimizing cost and materials wastage. In this study biodiesel was produced from hydrolysate (free fatty acids from hydrolyzed *Jatropha curcas* oil) using calcinated niobic acid catalyst at controlled rates of process parameters. Yield and conversion up to 97.7% and 100% respectively of the alkyl esters produced. This informs the influence of process parameters significantly on the throughput of the final product.

Keywords: Biodiesel; Hydrolysate.

Introduction

Biodiesel is a fuel of organic origin consisting of long chain fatty acids. The application of this fuel in diesel engine offers environmental benefits when compared with fossil fuel. Feed stocks of vegetable oil (Virgin and waste) and animal fats have been explored for production of biodiesel [1]. Other renewable resources from which biodiesel had been produced is algae and yellow lard [2]. Biodiesel is an environmentally friendly alternative liquid fuel that can be used in any diesel engine with little or no engine restructuring. Interest in organic oils consideration for biodiesel production has been kindled on account of its less polluting nature and its renewable source when compared with fossil diesel fuel [3]. Biodegradable fuels like biodiesels have an expanding range of potential applications as they are less environmental polluting. Therefore, there is growing interest in degradable diesel fuels that degrade more rapidly than petroleum fuels [4]. Biodiesel beckons increasing consideration from institutions and individual desire home brew biodiesel production.

The production of biodiesel from oil origin has utilized feed stocks from available biomass in the world over. For instance, in USA, the combined vegetable oil and animal fat production amounts to about 35.3 billion pounds per year [5]. Similarly, Brazilian production has witnessed predominant increase with about 2 million tons/year in 2009 utilizing about 60 plant oils [6]. Moreover, as at 2002, of the world production of biodiesel about 84% was from rapeseed oil, 13% from sunflower while palm oil and soybean biodiesel both record 1% [7] subject to the availability of these seed oil and the energy needs in the various locations of the world.

Transesterification primarily is the main catalyzed chemical reaction between triglycerides and alcohol to afford mono-esters [8]. The long and branched chain triglyceride molecules are transformed to monoesters and glycerin [9]. In transesterification, triglycerides are converted in sequential order to diglycerides and monoglycerides in reversible reactions. However, this approach is feasible if the FFA content is within the allowable limit. High FFA from hydrolyzed oil requires a prior esterification in biodiesel production.

The main vegetable oil materials of no food usage for biodiesel production are plant species such as *Jatropha* or *ratanjyote* or *seemaikattamankku* (*Jatropha curcas*), karanja or honge (*Pongamia pinnata*), nagchampa (*Calophyllum inophyllum*), rubber seed tree (*Hevca brasiliensis*), neem (*Azadirachta indica*), mahua (*Madhuca indica and Madhuca longifolia*), silk cotton tree (*Ceiba pentandra*), jojoba (*Simmondsia chinensis*), babassu tree, *Euphorbia tirucalli*, microalgae, *etc*. Their accessibility and easy growth benefits in various location and climates of the world as well as their relative affordability place them an edge over majority of oil food [10].

The non-food oils materials such as *Jatropha*, microalgae, neem, karanja, rubber seed, mahua, silk cotton tree, *etc*. are easily available in developing countries and support the economy of the developing nations commensurately when compared to oils used for food materials [11]. The oils from neem (*Azardirachta indica*) and rubber (*Hevea brasiliensis*) have high free fatty acid (FFA) content. The presence of FFAs in any feedstock materials put the material to danger of soap formation when an alkaline catalyst is use in the process. Moreover, there is also constraint of biodiesel-glycerol separation in the downstream process.

Jatropha curcas oil is a potential feed stock for biodiesel production. Apart from palm oil and algae no other biodiesel feedstock is capable of producing high yield of biodiesel as *Jatropha curcas* [12]. And as such, the food-energy feud of palm oil and the complexity involve in algae-oil generation among other constraints has intensify the improved interest in *Jatropha curcas* for consideration as feedstock material in biodiesel production.

The seed oil of *Jatropha* was utilized as a diesel fuel alternative

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during World War II and can as well serve as blends with conventional diesel [13,14]. Hence, the use of *Jatropha curcas* and *Pongamia pinnata (Karanja)* indicate a more suitable feedstock for synthesis of renewable fuel such as biodiesel [15,16]. The oil content of *Jatropha* and Karanja are very high reaching up to 30-50% [12,13].

Biodiesel is economically feasible in majority of oilseed-producing regions of the world [17]. Biodiesel is a technologically feasible substitute to petro-diesel, but biodiesel selling price doubled that of fossil diesel in most advance countries which is in part dependent on control measures of the reagent used in the production [17]. Although, biodiesel is still currently produced in relatively small scale compared to fossil fuel, present market price is not competitive. Hence, biodiesel at the present economic situation does not satisfactorily rivaled petro-diesel [17].

Commercially, biodiesel production is essentially same depending on whichever route is adopted in production. The process involves reaction of oil and fat with alcohol in the presence of a catalyst. The catalysts utilized over time have ranged from alkaline based catalyst (NaOH and KOH) which were used for commercial production. Others are heterogeneous base catalyst, acid based homogenous and heterogeneous catalyst as well enzyme catalyst [11].

Apart from the feedstock constituting the highest cost of biodiesel synthesis amounting to about 80% of the operating cost [18], the amount of other materials used in the process which depends largely on the control of process parameters such as temperature, catalyst dose, reaction time and molar ratio of alcohol used [19]. It is thus imperative that the amount or concentration of these parameters were determined in order to avoid wastage while also ensuring sufficient amount for optimum conversion and yield generation. This paper presents the finding of effect of process parameters on FAME yield and conversion using fluffy niobic acid calcined at 150°C.

Materials and Method

Calcination and characterization of powdered acid catalyst

The powdered niobic acid ($Nb_2O_5.H_2O$) catalyst utilized in this study was calcined in an indigenous "Iso Temp-220" furnace for duration of 4 hrs at 150°C. The white fluffy niobic acid powder was subsequently stored in the desiccator until needed for use. Characterization of the calcined catalyst was done using Fourier Transformed Infrared Spectroscopic (FT-IR) analysis.

Batch esterification reaction

The batch esterification reaction was carried out in 250ml screwed-cap shake flask and the content of the flask was made to react by monitoring the operating parameters in an incubator shaker (INFORs AG CH-4103 BOTTMINGEN). 16gm *Jatropha curcas* hydrolysate (FFAs produced by enzymatic hydrolysis of crude *Jatropha curcas* oil) was put in the flask with a catalyst loading varied between (1.0-5.0 wt% relative to the FFA) while variation of the methanol to oil molar ratio was between 3:1 to 7:1. The basis of methanol to oil ratio selection was relative to the molar weight of 819 g/mol of the oil. The reaction temperature was varied between 45-65°C and agitation rate ranges between 100-500 rpm to investigate the influence of the various tested parameters. The reaction was also monitored starting from 3-7 hrs to test the effect of time on the yield and conversion of the FFA.

After completion of every experimental run, the effluent was centrifuge in a (Rotina 38 Zentrifugen D-78532 Tuttlingen). The oil and unreacted methanol phase were decanted into a separating funnel and was left overnight to separate the oil and excess methanol. The final biodiesel product was incubated in an oven for 3-4 hrs to eliminate the moisture generated during the process. Each experiment was carried out in successive triplicate and estimation of biodiesel yield was based on the weight of biodiesel produced and weight of the FFA used in the reaction while the FFA conversion was due to the amount of initial FFA and the residual FFA as measure of the acid value [3] respectively.

The standard error of ±1-2% was obtained from the estimation of both the yield and conversion for the set of the experimental triplicates.

$$Yield\ (\%) = \frac{weight\ of\ biodielsel}{weight\ of\ FFA} \times 100 \qquad (1)$$

While the % conversion was determined by estimating the acid value after the reaction and was calculated by the equation below

$$C = \left(1 - \frac{Av_t}{Av_0}\right) \qquad (2)$$

Where C is the FFA conversion, Av_t=final acid value and Av_0= initial acid value before esterification reaction.

Results and Discussion

Fourier Transform Infra-Red Spectroscopy (FT-IR) is use for elucidation of structures of absorption, emission, photoconductivity or Raman scattering of substance [20]. It shows the absorption peaks of sample which corresponds to frequencies of variations between the bonds of the atoms of the material's constituents. The FT-IR of the powdered niobic acid catalyst after calcination is presented in Figure 1 below. The spectra of the calcined solid acid has broad band at 3500 cm^{-1} due to the O-H stretching mode of hexagonal groups [21]. Moreover, peak observed between 2850 cm^{-1} and 2900 cm^{-1} is attributed to asymmetric stretching of CH_3 group. The small peak observed close to 2400 cm^{-1} is attributed to C=O stretching vibrations of ketone, aldehyde, lactone and carbonyl group [22]. Peak observed at 1800 cm^{-1} also informs a C=O stretching vibrations of ketone and aldehyde groups relative to attached NH_2 groups.

Batch esterification reaction

Temperature effect: Study on the effect of temperature variation on the esterification of *Jatropha curcas* hydrolysate is as shown in Figure 2a. At the commencement of the reaction, higher rate was predominant relative to concentration of free fatty acids. Almarales et al. [23] reported a FAME conversion at 30 minutes reaction time at high temperature of 200°C. In this study, optimum FAME conversion and yield was favoured up to 60°C at 15% relative to the initial yield in the reaction. Higher temperature above 60°C lowers FAME yield in comparison with result of FAME conversion at 60°C. Comparable result of study carried out by Marchetti et al. [24] and Patil and Deng [25] show temperature effect to have positive contribution on fatty acids esters conversion up till 60°C which is in agreement with finding from the study. Thus, suffice to conclude that temperature above 60°C does not favour conversion of the FFA as boiling point of methanol is gradually reached.

Effect of oil-alcohol molar ratio: The effect of molar ratio of methanol to oil on FFA conversion and yield was investigated from 3:1 to 7:1. Figure 2b shows the influence of methanol on conversion of FFA and yield. Methanol-oil ratio of 5:1 offered the best combination for optimum conversion and yield. This finding is comparable with that reported by Almarales et al. [23] in hydroesterification of *Nannochloropsis oculata* microalgae' biomass to biodiesel using Al_2O_5 supported Nb_2O_5 catalyst. Increasing the methanol ratio further resulted in reduced conversion. Ramadhas et al. [8] reported that acid

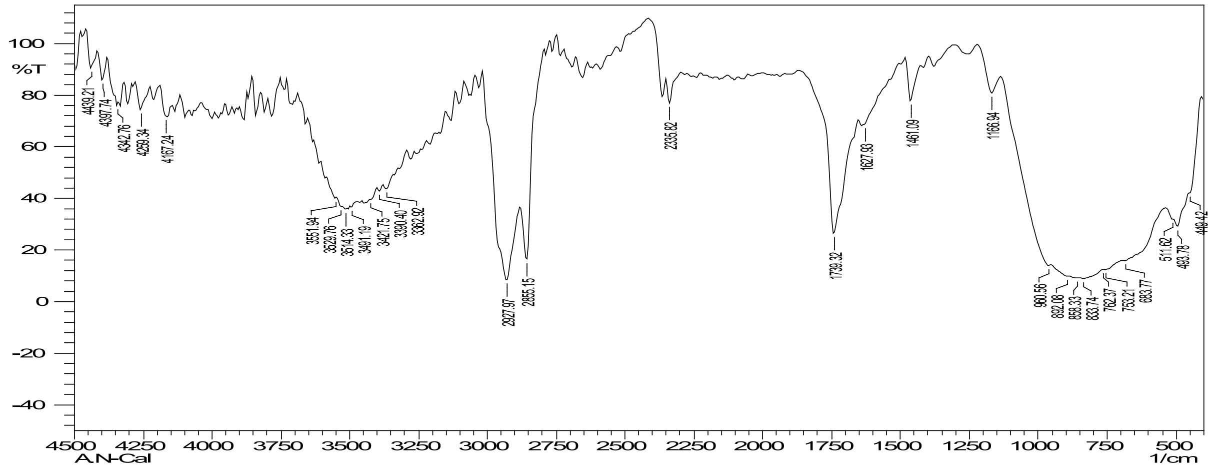

Figure 1: Ft-IR analysis of calcined powdered catalyst.

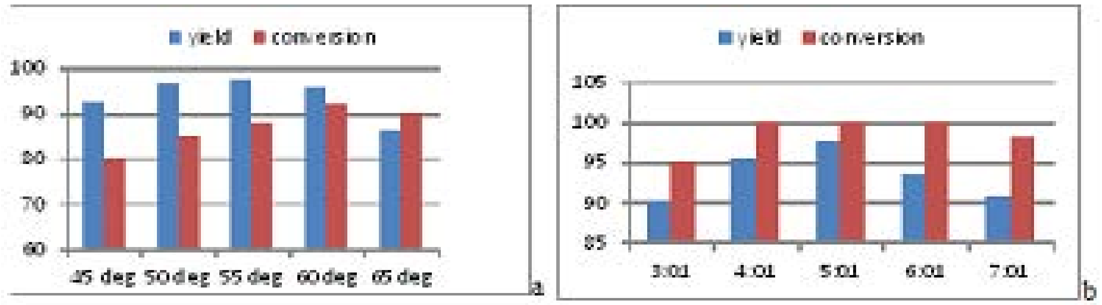

Figure 2a and 2b: Temperature and molar ratio effects on yield and conversion of FAME from FFA in %.

esterification reduces high FFA oil with high amount of methanol while also occurring at longer reaction time. Contrary to finding in this study lower methanol t oil ratio produced higher yield and conversion reaching 100% at 4:1 methanol to FFA ratio.

Effect of mixing rate: Agitation rate of 400rpm produced the effective conversion; nevertheless less magnitude was recorded at further increase on the agitation rate for the yield of the biodiesel and its FAME conversion (Figure 3a). This may be attributed to shift of reaction towards the reverse reaction. Agitation significantly affects the reaction rate; insufficient mixing could inhibit the reaction rate, which could lower the product formation.

Conversely, higher agitation impacts negatively on the reaction and higher energy is also expended while lower throughput is recorded. Thus the yield of biodiesel above 400rpm agitation rate was observed to be lowered as the evaporation of methanol causes insufficient contact with the substrate.

Effect of catalyst dosage: The powdered niobic acid catalyst dose was used to investigate FAME conversion and yield. The considered variation (1-5 wt%), optimum biodiesel yield was recorded at 4 wt% of the catalyst while the conversion continue to increase with 5 wt% of catalyst above 90% (Figure 3b). Carvalho et al. [26] reported that catalyst with acidic sites such as niobium oxide produce good conversion in esterification process. The catalyst is thermally stable and diffusion problem is reduced. Almarales et al. [23] obtained 92.24% conversion using Nb_2O_5 (Al_2O_3) and 87.43% using pure Nb_2O_5 at higher catalyst loading of 20% each. In this study a higher yield and conversion was achieved with lower catalyst dose. Comparable result was achieved by Umdu and Erol [27] in transesterification of *Nannochloropsis oculata* microalgae lipid to FAME with Al_2O_3 supported on CaO and MgO Catalyst with result such as that recorded in this study.

Reaction time effect: In this reaction step, conversion of the *Jatropha curcas* hydrolysate from to methyl esters was investigated over 7 hrs periods. The yield was proportional with the time increment while FAME conversion attained optimum value of 96% at 6 hrs reaction time. Figure 4 presents the effect of reaction time of FAME conversion and yield.

Srilatha et al. [20] studied the effect of reaction time on esterification of palmitic acid and sunflower fatty acid using heteropoly tungstate supported on niobia catalyst. Similar to the findings in this study FFA conversion was observed to increase with time. However lower reaction time (1-2 hrs) was achieved compared to 6 hrs optimum recorded in this study. This may be due to pure niobic acid used in the study compared to the heteropoly tungstate supported on niobia.

Conclusion

In the esterification reaction study to convert *Jatropha curcas* hydrolysate from hydrolyzed *Jatropha curcas* oil to biodiesel, we showed that parameters influence on yield and conversion rate were estimated. Optimum yield and conversion was determined to be 96% and 100% respectively at 5:1 methanol to FFA ratio, agitation of

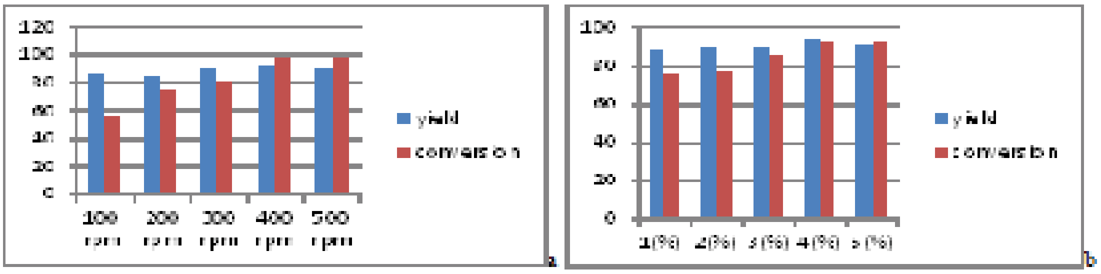

Figure 3a and 3b: Effect of mixing rate and catalyt dosage on FAME conversion and yield from FFA in %.

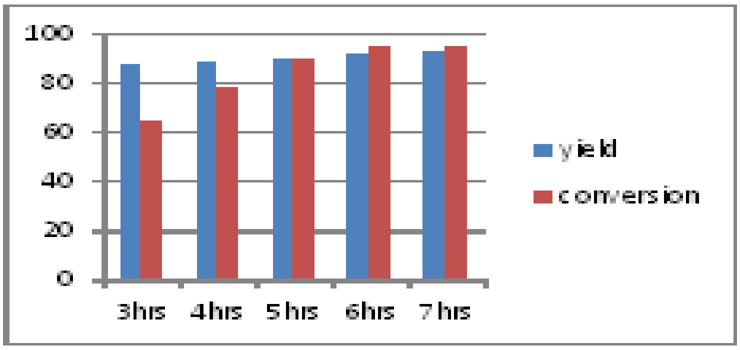

Figure 4: Effect of reaction time of FAME conversion and yield from FFA in %.

400rpm, catalyst loading of 4 wt%, temperature of 60°C and reaction time of 6 hrs. All the control parameters significantly affect FAME conversion while yield generation was less affected in comparison with the lowest control parameters from commencement of the reactions. It is thus sufficient to conclude that the control of the parameter best affect the conversion more than the yield as the FAME Conversion was observed to continually increase rapidly while the yield recorded lower or insignificant increase as the reactions proceeded.

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