



Effect of reaction temperature and time on neem methyl ester yield in a batch reactor



C.N. Anyanwu ^{a,*}, C.C. Mbajiorgu ^b, C.N. Ibeto ^a, P.M. Ejikeme ^c

^a National Centre for Energy Research and Development, University of Nigeria Nsukka, Nigeria

^b Dept. of Agricultural and Bioresources Engineering, University of Nigeria Nsukka, Nigeria

^c Dept. of Pure and Industrial Chemistry, University of Nigeria Nsukka, Nigeria

ARTICLE INFO

Article history:

Received 13 February 2013

Accepted 28 April 2013

Available online 2 June 2013

Keywords:

Biodiesel

Modelling

NLREG software

Transesterification

Neem oil

ABSTRACT

Experimental investigation of neem methyl ester yield in a batch reactor at different process conditions of temperature and reaction duration was carried out using a 2-factor, 5-level full factorial experimental design. Reaction temperature was varied between 40 °C and 60 °C, while reaction time was studied in the range of 30–120 min. The study, which was carried out using 1% w/w catalyst amount and alcohol to oil ratio of 6:1, showed that reaction temperature had a highly significant effect ($p < 0.01$) in comparison with reaction time, which had a significant effect ($p < 0.05$) on methyl ester yield. Six (6) empirical models were developed for the response variable of methyl ester yield using a non-linear regression analysis method, facilitated by NLREG version 6.3 software. Results showed that the mean predicted ester yield values and mean experimental values were not statistically different at the 95% confidence level, whereas the maximum deviation observed was 6.9%.

© 2013 Elsevier Ltd. All rights reserved.

1. Introduction

Global carbon dioxide emission is rising and has already exceeded the dangerously high level of 450 ppm [1]. This has led to serious concerns regarding on-going and impending climate change phenomena in many parts of the world, necessitating more efforts towards reducing the use of fossil fuels by increasing the application of renewable energy derived fuels, especially for transportation. Biofuels, particularly those derived from second generation biomass resources have been shown to be useful alternatives to conventional transport fuels. Biodiesel – a first generation biofuel – derived from vegetable oils and fats is a suitable alternative fuel for compression ignition engines. As an alternative fuel, biodiesel is becoming increasingly important due to diminishing petroleum reserves and adverse environmental consequences of exhaust gases from petroleum-fuelled engines [2]. The biodiesel manufacturing process converts oils and fats into mono alkyl esters, or biodiesel. These chemicals are also referred to as fatty acid methyl esters (FAMES).

Although straight vegetable oils (SVOs) have been applied to compression ignition (CI) engines as alternative fuels, starting with Rudolph Diesel's successful trials in the early 19th Century, when he used peanut oil to power his CI engine, research has shown that raw vegetable oils can cause long-term problems to the engine

owing to their high viscosity [3,4]. Among other methods of conversion [5], transesterification of vegetable oils into biodiesel has become a standard way of producing fuel grade products from them by reducing their viscosity to acceptable values of between 1.9 and 5.9 mm²/s. Biodiesel has numerous advantages over fossil-derived diesel fuels as reported elsewhere [3,4,6], but the major factor limiting its widespread utilization is high cost of production. The high production cost of biodiesel derives directly from the high price of vegetable oil raw materials, most of which are edible (first generation biofuel sources) and serve as food for man. Non-edible oils such as neem, jatropha and other oils are, therefore, becoming more important as biodiesel feedstock [2].

Apart from high vegetable oil prices, the processing cost of biodiesel is also high, constituting about 20–25% of total cost of production in the acid/base catalyzed methyl ester process. Heterogeneous catalysts are becoming increasingly important [7], but homogeneous catalysts have some advantages in large scale biodiesel production [8]. There is a large number of commercial plants producing biodiesel by transesterification based on base catalyzed (caustic) homogeneous transesterification of vegetable oils due to a number of advantages of such catalysts [7,9]. Modelling the ester yield as a function of process parameters could enable better estimation of biodiesel yield from the transesterification process and ultimately reduce the number of practical experiments required for process optimization and therefore, the production cost of biodiesel. Some process parameters that affect the ester yield during transesterification of vegetable oils include alcohol-to-oil ratio,

* Corresponding author. Tel.: +234 805 1980070.

E-mail address: cnasofia@gmail.com (C.N. Anyanwu).

catalyst amount (which varies between 0.5–1.5%), reaction duration and temperature. Other factors, such as degree of agitation, and nature of feedstock are also of importance [10].

Neem tree (*Azadirachta indica*) thrives very well in Nigeria and has indeed been applied as a desert control agent in parts of northern Nigeria and neighbouring West African countries. The tree grows naturally in areas with 450–1200 mm annual rainfall but has been introduced successfully even in areas where the rainfall is as low as 150–250 mm. Neem grows on altitudes up to 1500 m. It can grow well in wide temperature range of 0–49 °C and requires a lot of sunshine [11]. However, it cannot withstand water-logged areas and poorly drained soils. The pH range for the growth of neem tree lies in between 4 and 10. Neem trees have the ability to neutralize acidic soils by a unique property of calcium mining [12]. The oil is relatively abundant in Africa and portends great prospects for the development of agriculture. In spite of these prospects, there are few reports on the energy applications of neem oil, in comparison with other non-edible oil crops like jatropha.

The present research work is intended to contribute towards better understanding of the biodiesel process with neem oil feedstock. It dwells on the experimental investigation and empirical modelling of the influence of reaction temperature and duration on neem oil methyl ester yield at 1% catalyst amount and a 6:1 alcohol to oil ratio, using non-linear regression analysis. The ranges of reaction temperature and duration studied were chosen based on literature reports [13].

2. Materials and methods

The fatty acid profile of the neem oil was first studied using Agilent 7890A Series Gas Chromatograph with split/splitless injector, Agilent Technologies (Stevens Creek Blvd., Santa Clara, CA, United States). Results of the chromatographic analysis, presented in Table 1, indicate that the molecular mass of the oil is 871.992. It also showed that the neem oil feedstock was about 99% pure.

The reactor used to carry out the experiments is a 50 L capacity batch reactor (Fig. 1) designed and fabricated for the study with a cylindrical section (37 cm diameter and 48 cm height), which tapers into a lower conical section for ease of product evacuation [3]. The reactor was constructed using stainless steel plate of 2 mm thickness and lagged with glass wool insulator, whereas a simple flat bar of cast iron was used to fabricate the agitator paddle. The heat supply source was a 2.5 kW electrical coil, controlled by a linear thermoregulator based on LM 35 integrated circuit sensor, which was developed for the study. The thermoregulator was calibrated in neem oil environment against the readings of a K-type thermocouple, BK Precision (Made in Taiwan). The top-entering agitator for the reactor was driven by a 746 W (1 Hp) single phase electric motor, mounted on the reactor stand and supported on a ball bearing to reduce wobbling effect. Agitation speed was maintained at 1350 rev/min or 2.5 rev./s for all the experiments.

Table 1
Fatty acid profile of neem oil. Source: [10]

S. no.	Fatty acid description	Fatty acid name	Composition (%)	^a Range of composition (%)
1	C16:0	Palmitic acid	16.65	16–33
2	C16:1	Palmitoleic acid	0.24	Trace
3	C18:0	Stearic acid	18.39	9–24
4	C18:1	Oleic acid	46.65	25–54
5	C18:2	Linoleic acid	15.41	6–16
6	C18:3	Linolenic acid	0.56	Trace
7	C20:0	Arachidic acid	1.04	Trace
		Total	98.94	

^a Molecular weight = 871.992.

Neem oil used for the present work was purchased from a farm in Katsina State of Nigeria and pretreated via degumming and acid esterification following a procedure described by Ramadhas et al. [14]. 20 L of the pretreated neem oil with water content of 0.20% and free fatty acid (FFA) content of 0.13% was measured out and transferred into the reactor. The thermoregulator sensor was inserted into the reactor, while the agitator was connected to a power supply source.

3. Experimental

The pre-determined quantity of NaOH, corresponding to 1% w/w of the oil was mixed separately with the methanol (in a 6:1 alcohol to oil ratio) and stirred to dissolve it. Although some researchers favour the use of 1.5% catalysts amount in the base catalyzed process [15], others such as Vincente et al. [10] have reported nearly 100% biodiesel yield while using 1% catalyst. The reactor content was first preheated to the desired temperature (at which the thermoregulator was set) according to the experimental plan. When the neem oil had attained the desired temperature and the catalyst completely dissolved, the methanol/NaOH mixture was poured into the oil and the reactor closed tightly. At the end of each reaction, the content of the reactor was transferred into a tall glass cylinder for gravity separation of the glycerol and biodiesel. The supernatant phase (FAME) was carefully separated and washed several times with tap water until a neutral pH value (of 7.0) was attained. The quantity (volumetric yield) of the FAME was measured and converted to percentage yield with respect to the oil. A summary of ester yields (EY) obtained for different treatment combinations (experimental points) is presented in Table 2.

The experimental plan applied in this study is a 2-factor, 5-level factorial design. The factors are reaction duration (time) and temperature. Reaction duration was varied between 30 min and 120 min with a step of 22.5 min, while temperature was studied in the range of 40–60 °C, with a step of 5 °C. To ensure accurate measurement of reaction temperature, a linear thermoregulator was developed based on the LM35 integrated circuit (as described by Anyanwu et al. [3]) and used in this work. A total of 25 treatment combinations were studied, at which transesterification reaction was carried out (Table 2).

To determine the quantity of FAME after each experiment, FAME sample was derivatised using t-butyltrimethylchlorosilane. A 1% heptadecanoic methyl ester (HME) in hexane was prepared and HME equivalent to 5% of the total FAME was added to each FAME dilution as internal standard. This mixture was analyzed using Agilent 7890 GC equipped with FID Detector, Agilent 7683 series Injector and Agilent 19091N Column of length 30 m and internal diameter 0.32 mm.

The experimentally determined values of ester yield were presented graphically to enhance understanding of the nature of the curves and, thus facilitate the regression modelling (curve fitting) aspects of the work. Thereafter, NLRG version 6.3 software was used to develop regression models expressing the dependence of

Download English Version:

<https://daneshyari.com/en/article/7166640>

Download Persian Version:

<https://daneshyari.com/article/7166640>

[Daneshyari.com](https://daneshyari.com)