**Optimization of Acid Catalyzed Transesterification of Jatropha and Rapeseed Oil with 1-Butanol**

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**ABSTRACT:**

The acid catalyzed transesterification of rapeseed and jatropha oil with 1-butanol was studied by varying temperature, reaction time, butanol/triglyceride ratio and the amount of sulfuric acid. A full factorial design supporting non-linear regression models was used to combine the four design parameters. The measured yield, viscosity and acid number were quantitatively described by PLS-regression as functions of the four design variables for each vegetable oil. Product purity was controlled by 1H-NMR. The PLS-regression model was used for optimizing the three response parameters. High (100%) yield is easily obtained within the experimental domain, whereas viscosity and acid number, with different optimum conditions, have to be compromised in order to keep both as low as possible. While rapeseed reached optimum within the experimental limits, the results indicate that for jatropha further reduction in viscosity and acid number requires increasing temperature and/or the butanol/triglyceride ratio beyond the upper limits of the design.

**Keywords**

Biodiesel

Fatty acid butyl ester

FABE

Factorial design

PLS-regression

Response surface

**1. Introduction**

First generation biodiesel is usually made by base catalyzed transesterification of triglycerides with methanol. The result is fatty acid methyl esters (FAME). The triglyceride feedstock in Europe is most commonly rapeseed oil, but can be any of a range of oil-bearing crops and also animal fat or used cooking or frying oil [1, 2]. Non-edible oils like oil from *Jatropha Curcas* are considered as second generation feedstock [3]. Jatropha oil is non-edible due to poisonous substances. Furthermore, it can be grown in dry wastelands and sandy soil [3, 4].

The main reason for the transesterification is to produce a fuel with acceptably low viscosity. Using vegetable oils directly in diesel engines may lead to for example carbon deposits, injector plugging and piston sticking [4-8]. Alternatives like blending the vegetable oil or forming micro emulsions give the same engine problems, while transformation to low-viscosity fuel through controlled pyrolysis is very expensive [5].

To make the biodiesel 100% renewable, the methanol should originate from biomass or biogas (methane) or should be substituted with ethanol or butanol which may be produced by fermentation [6, 9]. A benefit from substituting methanol with other short-chain alcohols is improved cold flow properties and reduced cloud point temperatures [10, 11]. Butanol is an attractive bio-fuel, and extensive research is put into the further development of fermentation processes for competitive production of bio-butanol [12, 13].

The transesterification can be performed either with an acid or a base catalyst. However, the alkali catalysts do not perform well with the larger alcohols [6, 7]. Methanol (pKA=15.09) readily form methoxide, which is the reactive specie. Larger alcohols like 1-butanol (pKA=16.1) [14] remains mostly protonated in contact with water (pKA=15.74) or the hydroxide catalyst. The acid-catalyzed reaction on the other hand begins with protonation on one carbonyl-group of the triglyceride; and is less sensitive to the acid/base properties of the alcohol, although larger alcohols like 1-butanol are reported to react better than methanol [11]. The acid-catalyzed reaction also works well with oils containing significant amounts of free fatty acids (FFA) which undergo acid-catalyzed esterification resulting in the same esters as the transesterification. Feedstock oils not suitable as food, like waste frying oil, non-edible oils or algae oils typically contain significant volumes of FFA [4, 8, 11, 15]. For example, seeds from jatropha contain approximately 50% oil, mostly triglycerides, but including approximately 14% FFA. A drawback with the acid-catalyzed process is the need for elevated temperatures. Sulfuric acid is mostly used as catalyst, but also phosphoric acid and hydrochloric acid among others are reported [4-7, 11].

The purpose of the present study was to explore the acid-catalyzed transesterification of jatropha and rapeseed oil with 1-butanol by varying temperature, reaction time, the butanol/triglyceride ratio, and the amount of sulfuric acid. Statistical experimental design was used to combine the four design parameters, and partial least squares (PLS) regression was used to quantitatively describe measured yield, viscosity and acid number as functions of the four design variables for each of the vegetable oils.

**2. Materials and Methods**

*2.1 Materials*

Jatropha oil was from Terazol Energy, India. Acid number was 10.29 mgKOH/g and viscosity 38.8 mm2/s (34.95 cP and 0.902 g/mL at 40 °C). Crude degummed rapeseed oil was from Mestilla, Litheunia. Acid number was 2.54 mgKOH/g and viscosity 36.7 mm2/s (33.23 cP and 0.904 g/mL at 40 °C). The methods used to determine acid number and viscosity are described below. 1-Butanol and concentrated sulfuric acid were of analytical grade from Merck KGaA.

*2.2 Experimental design*

Based on the cited literature, temperature (Temp), reaction time (Time), the butanol/triglyceride ratio (Al/Tr), and the amount of sulfuric acid (Cat) were selected as design parameters and varied with the following ranges: 70-110 °C, 1-3 hrs, butanol/triglyceride ratios of 4-8 equivalents, and 1-3 v/v% sulfuric acid. A pre-study with 12 experiments was performed with jatropha oil to verify reasonable parameter ranges. The main experiments were designed as full factorial designs [16] (Central Composite Face, CCF) supporting multivariate regression models with quadratic and cross terms (response surface modeling). The design resulted in 24 individual experiments plus replicated center point giving a total of 28 experiments. The experimental design is shown in Table 1. Run order was randomized.

*2.3 Transesterification*

Transesterifications were carried out in 50 mL round bottom flasks which were placed in a Starfish 5-well monoblock from Radleys, fitted with inserts for 50 mL flasks on top of an IKAmag RCTbasic hotplate with stirring. The temperature was controlled with an IKAtron ETS-D4fuzzy temperature controller to ±1 °C, where the temperature probe was placed in one of the round bottom flasks filled with equal volume of oil and butanol. Stirring was provided with 2 cm egg shaped PTFE-coated magnets at high speed providing homogenous solution at all times. Two flasks were used for each experiment allowing two experiments to be performed in parallel. As preparation the desired amount of butanol was filled in one flask, and triglyceride in the other. The setup was heated to the desired temperature which was kept constant. Sulfuric acid (in vol% of the triglyceride volume of 25-30 mL) was added to the butanol (in mol/mol equivalents in relation to the triglyceride volume), and given a short time to reach the desired temperature. The mixture of butanol and sulfuric acid) was added to the triglyceride at reaction start.

After the desired reaction time the flask was cooled under running tap water (5-8 °C) and the reaction mixture was poured into a separation funnel with 50 mL of tap water, and mixed gently to avoid formation of emulsions. The first washing required 15 min of settling time before the water phase could be removed. Two more washings with 50 mL water required less settling time. The butanol and most of the glycerol were removed on a rotary evaporator before the final remains were removed by stirring under 0.16-0.20 mbar pressure before the neat biodiesel was weighed and analyzed.

*2.4 Yield calculations*

Yield was calculated from the initial triglyceride weight and the final weight of the produced FABE. Based on the relative composition of fatty acids as reported by Hoekman et al. [2] the average molecular weights of the rapeseed and jatropha oils were calculated to 864 and 874 g/mol, respectively. The average molecular weights were 331.33 and 334.67 g/mol for FABE from rapeseed and jatropha, respectively.

*2.5 Viscosity measurements*

The dynamic viscosity was measured in centipoise on a Physica MC200 rheometer with application US200 version 2.3 software and measuring system MK24 (75 mm, 1°) at 40 °C. The rheometer measured 6 points between 100 s-1 and 1000 s-1 shear rate and calculated an average. All FABE samples displayed Newtonian fluid behavior within this range. The density was measured in an Anton Paar DMA-4500 at 40 °C. The kinematic viscosity in mm2/s (cSt) was calculated from dynamic viscosity and density.

*2.6 Acid number measurements*

The titrations were based on EU standard EN14104:2003, but modified to handle the small samples from the experiments: A 0.055 M KOH solution was prepared from KOH-pellets and 2-propanol and the concentration was standardized each day against 0.050 g benzoic acid, with bromothymol blue as indicator ( 5 measurements). Each FABE sample (4 g) was dissolved in 2-propanol (50 mL) with phenolphthalein (0.15 mL) as indicator and titrated. The acid number was calculated from the average of 3 titrations.

*2.6 PLS-regression*

PLS-regression [17] was used to quantitatively relate the three response variables yield, viscosity (Visc) and acid number (AN) with the four design variables for each oil. Prior to PLS-regression, the data were mean centered and scaled to unit variance. The PLS models were validated by explained variance, and goodness of fit (R2) and prediction (Q2), the latter obtained after cross validation [18]. Modde 10 from Umetrics, Umeå, Sweden, was used for the experimental design and the PLS regression.

**3. Results and Discussion**

High yield was obtained in all experiments with a few exceptions. The lowest yield was 93.6% and 92.7% for jatropha and rapeseed, respectively, as shown in Table 1. The reproducibility between the four replicates (experiments 25-28) was very good.

PLS-regression of the data in Table 1 resulted in PLS models with generally high values for goodness of fit (R2) and prediction (Q2), particularly for yield and viscosity, as shown in Table 2. Fig. 1 shows the observed versus predicted values for the three response variables for both jatropha and rapeseed. Table 1 and Fig. 1 illustrate that the three experiments carried out at the lowest temperature, number 1, 5, and 9, produced FABE with high viscosity, mostly due to unreacted triglyceride (this was verified with 1H-NMR analysis as described in supplemental material). Samples with 100% yield showed no trace of triglycerides (<2%). Contrary to the methanol/FAME process we did not get any phase separation with butanol/FABE. Thus unreacted triglyceride and FFA will follow the product and be included in the yield, although with a lower mass than FABE. Esterification of FFA also contributes to increased yield.

However, samples 1, 5 and 9 are well explained and predicted, implying that there is no reason to remove them from the model. These three experiments with high viscosity resulted in relatively low yield. The inverse relationship between yield and viscosity is also illustrated by the PLS-regression coefficients in Fig. 2 which also illustrates that temperature, time, and the butanol/triglyceride ratio have strong impact on yield and viscosity (positively and negatively correlated, respectively). The amount of catalyst has minor impact on yield but affect viscosity. On the other hand, acid number is positively correlated with the amount of catalyst (sulfuric acid) and negatively correlated with the butanol/triglyceride ratio. Acid number is relatively insensitive to reaction temperature and time.

The PLS models are generally improved by the interaction and particularly the square terms, although these terms are generally not really significant as can be seen by the relatively large confidence intervals. There were small differences between the models for jatropha and rapeseed oils.

The PLS-regression models describing the three response parameters as functions of the four predictor variables for each of the oils can be used to optimize the process. The optimum conditions imply high yield and low viscosity and acid number. According to the American and European standards (ASTM D6751 and EN 14214) viscosity should be below 6.0 mm2/s or 5.0 mm2/s, respectively. The acid number should be below 0.5 mg KOH/g. Table 3 shows the results of the optimization of the regression models. The table shows the results of an optimization when compromising viscosity and acid number, and in addition when prioritizing minimum viscosity or acid number. Maximum yield of 100% is easily obtained, whereas viscosity and acid number approach the target values. It is emphasized that the optimization is carried out within the constraints of the experimental design. According to the table, optimization requires temperature and Al/Tr at maximum design values for jatropha and slightly below the maximum levels for rapeseed, whereas time and catalyst are at intermediate and even lower levels. This implies that further reduction in viscosity and acid number requires increasing temperature and/or Al/Tr beyond the upper values used in the present design. Jatropha, containing more FFA, require more catalyst and alcohol compared to the rapeseed oil. Extrapolation of the model beyond the valid parameter range suggests that temperatures above the boiling point of butanol (117 °C) would be beneficial i.e. give both lower viscosity and acid numbers, particularly for jatropha. Pressurized reactors might make it possible to achieve both viscosity and acid number below the biodiesel requirements from pure FABE. Bouaid *et al*.[19] recently reported that the kinematic viscosity of FABE from rapeseed oil was 4.89 mm2/s (base catalyst). FABE from jatropha is reported to have 6.74 mm2/s (acid catalyst) [20].

The optimization of viscosity and acid number for jatropha is illustrated in Fig. 3. The response surface plots are made to illustrate one response at a time as function of two design parameters, keeping the two other design parameters at constant levels. Temperature and the butanol/triglyceride ratio are kept constant at the maximum design levels which are also the optimum levels (Table 3) whereas time and catalyst concentration are varied within the design ranges. The response surface plots illustrate that each of the response variables viscosity and acid number can be minimized, however, not simultaneously. As a consequence, optimum conditions have to be a compromise between acid number and viscosity as shown in Table 3. At the highest temperatures the viscosity seems to increase at excessive reaction time and/or catalyst concentration as can be seen in Fig. 3. Some of this increase may be due to an “over-cooking” effect where the double-bonds of the fatty acids are isomerized from *cis* to *trans* by acidic catalysis. Methyl esters of fatty acids with *trans* double-bond configuration is known to have higher viscosity than the corresponding *cis* compounds [21, 22], and a similar effect must be expected for the butyl esters.

The model for rapeseed oil is quite similar but reaches its optimum at a temperature and a butanol/triglyceride ratio slightly below the maximum design levels (Fig. 4). The different FFA contents should be important for these models, although there are also differences in the fatty acid composition between rapeseed and jatropha oils.

**4. Conclusions**

Factorial design and PLS-regression was used in prediction and optimization of the three response parameters in relation to the four predictor variables. High (100%) yield is easily obtained within the experimental domain, whereas viscosity and acid number, with different optimum conditions, have to be compromised in order to keep both as low as possible. While rapeseed reached optimum within the experimental limits, the results indicate that for jatropha further reduction in viscosity and acid number requires increasing temperature and/or the butanol/triglyceride ratio beyond the upper limits of the design.

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**Appendix A. Supplemental material**

NMR methodology and results.

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**Figure Captions**

**Fig. 1.** Observed versus predicted response variables for the PLS regression models. Experiment numbers correspond to numbers in Table 1. a) Jatropha oil, b) Rapeseed oil.

**Fig. 2.** PLS regression coefficients for the design variables in the model. a) Jatropha oil, b) Rapeseed oil.

**Fig. 3.** Optimization of viscosity and acid number for jatropha as function of time and catalyst (temperature constant at 110°C and Al/Tr at 8, i.e. maximum levels).

**Fig. 4.** Optimization of viscosity and acid number for rapeseed as function of time and catalyst (temperature constant at 108.7°C and Al/Tr at 7.2).

**Table 1**

Experimental design and measured values for the experiments.

|  |  |  |
| --- | --- | --- |
| Experimental design | Jatropha oil | Rapeseed oil |
| Exp. No. | Temp°C | Timeh | Al/Trmol/mol | Catv/v % | Yield% | Viscmm2/s | ANmg KOH/g | Yield% | Viscmm2/s | ANmg KOH/g |
| 1 | 70 | 1 | 4 | 1 | 94.6 | 12.91 | 2.82 | 94.5 | 12.94 | 2.67 |
| 2 | 110 | 1 | 4 | 1 | 97.5 | 7.64 | 1.86 | 98.9 | 6.68 | 1.44 |
| 3 | 70 | 3 | 4 | 1 | 96.1 | 9.19 | 1.82 | 98.0 | 7.43 | 1.32 |
| 4 | 110 | 3 | 4 | 1 | 98.4 | 6.59 | 1.57 | 99.2 | 6.40 | 1.34 |
| 5 | 70 | 1 | 8 | 1 | 97.1 | 13.05 | 1.18 | 97.2 | 11.39 | 0.60 |
| 6 | 110 | 1 | 8 | 1 | 99.9 | 6.25 | 1.18 | 99.7 | 6.29 | 0.92 |
| 7 | 70 | 3 | 8 | 1 | 99.4 | 7.70 | 1.15 | 99.1 | 7.53 | 0.83 |
| 8 | 110 | 3 | 8 | 1 | 101.0 | 6.43 | 1.25 | 100.6 | 6.25 | 0.94 |
| 9 | 70 | 1 | 4 | 3 | 93.6 | 11.69 | 2.97 | 92.7 | 12.51 | 2.67 |
| 10 | 110 | 1 | 4 | 3 | 98.7 | 6.34 | 2.27 | 99.7 | 6.46 | 2.73 |
| 11 | 70 | 3 | 4 | 3 | 97.3 | 7.19 | 2.28 | 98.0 | 6.90 | 1.78 |
| 12 | 110 | 3 | 4 | 3 | 100.0 | 6.96 | 4.04 | 100.0 | 6.62 | 3.47 |
| 13 | 70 | 1 | 8 | 3 | 95.4 | 8.16 | 1.83 | 96.3 | 8.01 | 1.28 |
| 14 | 110 | 1 | 8 | 3 | 99.7 | 6.30 | 1.51 | 98.3 | 6.29 | 1.93 |
| 15 | 70 | 3 | 8 | 3 | 99.1 | 6.62 | 2.28 | 98.8 | 6.36 | 1.30 |
| 16 | 110 | 3 | 8 | 3 | 99.5 | 6.09 | 1.77 | 99.9 | 6.37 | 1.60 |
| 17 | 70 | 2 | 6 | 2 | 97.2 | 7.67 | 1.38 | 98.3 | 7.26 | 1.10 |
| 18 | 110 | 2 | 6 | 2 | 100.0 | 6.43 | 1.83 | 100.2 | 6.11 | 1.19 |
| 19 | 90 | 1 | 6 | 2 | 99.4 | 6.95 | 1.43 | 98.9 | 6.38 | 1.02 |
| 20 | 90 | 3 | 6 | 2 | 100.0 | 6.39 | 2.10 | 99.9 | 6.09 | 1.39 |
| 21 | 90 | 2 | 4 | 2 | 98.3 | 6.78 | 1.71 | 98.5 | 6.34 | 1.49 |
| 22 | 90 | 2 | 8 | 2 | 100.0 | 6.46 | 3.02 | 99.1 | 6.17 | 1.03 |
| 23 | 90 | 2 | 6 | 1 | 99.7 | 6.71 | 1.95 | 99.9 | 6.22 | 0.65 |
| 24 | 90 | 2 | 6 | 3 | 99.5 | 6.36 | 2.72 | 99.4 | 6.10 | 1.60 |
| 25 | 90 | 2 | 6 | 2 | 99.7 | 6.13 | 2.08 | 99.4 | 6.27 | 1.37 |
| 26 | 90 | 2 | 6 | 2 | 99.7 | 6.11 | 2.17 | 99.1 | 6.25 | 1.41 |
| 27 | 90 | 2 | 6 | 2 | 100.0 | 6.09 | 1.93 | 98.4 | 6.22 | 1.50 |
| 28 | 90 | 2 | 6 | 2 | 99.6 | 6.14 | 2.21 | 99.3 | 6.15 | 1.41 |

**Table 2**

R2 and Q2 for the PLS-regression models

|  |  |  |
| --- | --- | --- |
|  | Jatropha oil | Rapeseed oil |
|   | Yield | Visc | AN | Yield | Visc | AN |
| R2 | 0.96 | 0.93 | 0.72 | 0.92 | 0.93 | 0.83 |
| Q2 | 0.76 | 0.63 | 0.10 | 0.62 | 0.68 | 0.45 |

**Table 3**

Optimum conditions for the different FABE properties.

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
|  |  | Temp | Time | Al/Tr | Cat | Yield | Visc | AN |
| Jatropha | Min. AN | 110.0 | 1.0 | 8.0 | 1.4 | 100.2 | 6.32 | **0.87** |
|  | Min. Visc | 109.6 | 1.9 | 6.9 | 2.3 | 100.6 | **5.26** | 1.74 |
|  | **Optimized** | **110.0** | **1.0** | **8.0** | **1.5** | **100.2** | **6.19** | **0.87** |
| Rapeseed  | Min. AN | 93.0 | 2.0 | 8.0 | 1.0 | 99.8 | 6.74 | **0.60** |
|  | Min. Visc | 110.0 | 2.0 | 6.0 | 2.0 | 100.4 | **5.34** | 1.42 |
|  | **Optimized** | **108.7** | **1.8** | **7.2** | **1.1** | **100.2** | **5.83** | **0.73** |

|  |  |  |  |
| --- | --- | --- | --- |
| a) | Yield | Viscosity | Acid number |
|  |  |  |  |
| b) | Yield | Viscosity | Acid number |
|  |  |  |  |

**Fig. 1.** Observed versus predicted response variables for the PLS regression models. Experiment numbers correspond to numbers in Table 1. a) Jatropha oil, b) Rapeseed oil.

|  |  |  |  |
| --- | --- | --- | --- |
| a) | Yield | Viscosity | Acid number |
|  |  |  |  |
| b) | Yield | Viscosity | Acid number |
|  |  |  |  |

**Fig. 2.** PLS regression coefficients for the design variables in the model. a) Jatropha oil, b) Rapeseed oil.

|  |  |
| --- | --- |
| a) Viscosity | b) Acid number |
|  |  |

**Fig. 3.** Optimization of viscosity and acid number for jatropha as function of time and catalyst (Temperature constant at 110°C and Al/Tr at 8, i.e. maximum levels).

|  |  |
| --- | --- |
| a) Viscosity | b) Acid number |
|  |  |

**Fig. 4.** Optimization of viscosity and acid number for rapeseed as function of time and catalyst (Temperature constant at 108.7 °C and Al/Tr at 7.2).

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