

INVESTIGATION OF PROCESS YIELD IN THE TRANSESTERIFICATION OF COCONUT OIL WITH HETEROGENEOUS CALCIUM OXIDE CATALYST

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ABSTRACT

The commercial success of biodiesels has to date been limited by high production costs of vegetable oil methyl esters. High feedstock costs are compounded by side reactions such as soap formation during conversion using conventional catalysts, and the consequent costs of product refining and purification. Recent studies on the heterogeneous catalysis of the transesterification reaction with commercial ion exchange resins have met with some success; the findings suggest that use of heterogeneous catalyst improves yield compared to conventional processing with homogeneous acid or alkaline catalyst. This study investigated the performance of heterogeneous calcium oxide catalyst in the production of coconut methyl ester. Specifically, the study investigated the effect of temperature, time, excess methanol and catalyst to oil ratio on conversion of oil in batch reactions as well as the level of trace calcium in the final product using a two-level factorial experimental design. The tests achieved conversion levels of 91.5 – 95.7%, based on measured TG levels of 0.6 – 1.2%. The specific gravity of the biodiesel phase was also found to be in the range of 0.83 – 0.87, which is also indicative of high conversion. Only temperature was found to have a statistically significant effect on triglyceride conversion, which implies that the overall rate of reaction is controlled by surface reaction kinetics rather than mass transfer. On the other hand, none of the experimental factors were found to have a statistically significant effect on the level of calcium contamination of the biodiesel product.

Keywords: Biodiesel; Calcium Oxide; Heterogeneous Catalyst

1.0 INTRODUCTION

Many countries have started to implement measures to reduce adverse impacts on their economies through energy efficiency and alternative fuel programs. The transportation sector has been affected more than other sectors due to its heavy dependence on petroleum products. Alternative fuels such as biodiesel have been proposed as substitutes to reduce the vulnerability of net oil importers. In addition, such fuels are expected to yield significant environmental benefits such as the reduction of emissions of greenhouse gases and air pollutants. Potential feedstocks for biodiesel include soya oil, rapeseed oil, coconut oil, palm oil, and jatropha oil, with the choice being dependent on the region.

Biodiesel is now available commercially in limited quantities, but its acceptance has been hindered due to its high cost as well as feedstock supply limitations. Global production of vegetable and marine oils in recent years was about 100 million t/a [1], which is mostly dedicated to traditional uses. Sharma and Singh [2] estimate the combined biodiesel potential of the top ten producing countries in the world at about 40×10^9 l/a, with production costs ranging from US\$0.5 – 1.7/l; significantly, they list three Southeast Asian countries among the top ten: Malaysia, Indonesia and the Philippines. In terms of volume, soya and palm

oil have the largest potential, while other vegetable oils such as corn and coconut oil are available in smaller quantities. Non-traditional feedstocks such as *Jatropha curcas* and oil-bearing algae are also the subject of much research interest [1-6].

With currently available raw materials and process technology, the main reasons for the high cost of biodiesel are:

- Absence of economies of scale in the emerging biofuels industry;
- High feedstock costs;
- Side-reactions, such as the saponification reaction, leading to feedstock losses and increased utility demands for product refining.

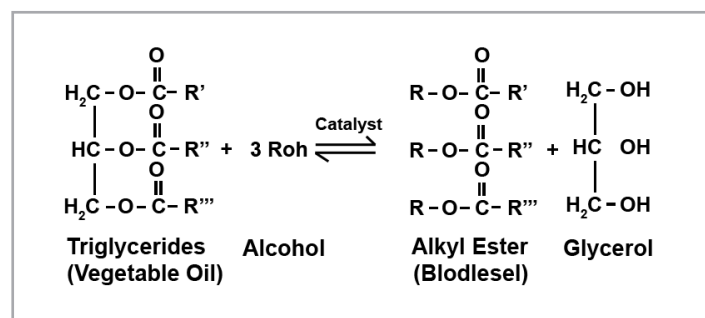
Improvements in process technology can potentially address the third factor. In principle, vegetable oils can be converted into biodiesels that closely approximate the properties of petroleum diesel by means of transesterification reactions. Theoretical details are discussed in the next section. By far the most commonly used means of making transesterification reactions proceed at commercially useful rates is through the use of alkali catalysts such as NaOH [1-5]. Their main drawback is that they react with naturally occurring free fatty acids to form soaps, which contaminate the methyl ester product and thus require subsequent

processing to yield a fuel product of acceptable quality. Thus, the use of alkaline catalysts may also require pretreatment of the feedstock oil to remove the fatty acids.

Acid catalysts are more suitable for highly acidic feedstocks; however, acid catalysed transesterification reactions proceed very slowly, and thus require larger, more expensive reactors for a given rate of production [4]. There is considerable interest in the use of highly selective enzymes of biocatalysts; however, the commercial potential of biocatalysis remains limited by the cost of the lipase enzymes. Another catalyst-free alternative requires the use of supercritical methanol at temperatures in the range of 200 – 400 °C. Supercritical plants can achieve high product yields even with low-grade feedstocks, but they entail high initial costs and are energy-intensive.

2.0 FUNDAMENTAL ASPECTS OF THE TRANSESTERIFICATION REACTION

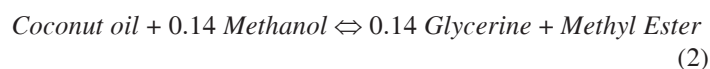
Vegetable oils consist primarily of triglyceride molecules with three fatty acid chains linked to a glycerol backbone. Naturally occurring oils also contain trace impurities, including diglycerides, monoglycerides, free fatty acids and other organic compounds. Depending on the precautions taken during the production and storage of vegetable oils, these impurities may be present in significant amounts which require downstream process interventions. For example, for edible oil production, free fatty acids are normally removed by neutralisation with NaOH and trace organic compounds with disagreeable flavors or colors are removed by adsorption and vacuum steam distillation. The transesterification reaction for converting triglycerides into methyl esters is



(1)

This conversion is necessary if the final product is to be used as engine fuels. Triglyceride molecules are much larger than hydrocarbon molecules found in diesel fuel; hence, vegetable oils are more viscous and less volatile than commercial diesel fuel. Such properties lead to poor combustion and fouling build up in the fuel injectors of diesel engines. Conversion to methyl ester is necessary to yield a product with smaller molecules, and properties more closely resembling those of diesel.

Note that three moles of methanol are needed for each mole of vegetable oil feed. However, since the reaction is reversible, excess methanol is normally used to shift the reaction forward. Furthermore, in the case of coconut oil whose molecular weight typically is in about 660 g/mole [7], the balanced reaction, expressed in theoretical mass ratios, is:



Hence, each gram of coconut oil is expected to yield one gram of methyl ester. The glycerol separates spontaneously from the biodiesel phase after the reaction. The latter phase contains the methyl ester as well as unreacted oil, whose molecular glycerol content can then be used to assess the extent of the reaction (pure methyl ester should be glycerol-free). Majority of excess methanol present dissolves in the glycerol phase.

The reactions given in Equations 1 and 2 take place in the presence of a catalyst, or at high pressures and temperatures, as discussed in the previous section. The presence of impurities, however, leads to undesirable side reactions yielding products that require further cleaning and refining. For example, free fatty acids in the feedstock oil react with alkaline catalysts to give soap. The soap must then be removed from the methyl ester product by a series of water- and energy-intensive washing operations. Such side reactions impose three process cost penalties: first, a portion of the raw material is converted to an undesired product; second, additional processing effort and cost are incurred to remove the undesired product; finally, the side reaction consumes some of the catalyst that is needed for the main process reaction. Thus, if a heterogeneous catalytic system can be developed such that these side-reactions are avoided, then substantial cost savings may be realized.

Glycerol is the main byproduct of the transesterification process. Its presence in biodiesel is highly undesirable as it can lead to engine damage; on the other hand, there is a considerable market for glycerol as a chemical commodity in its own right but the refined glycerol has a much higher price than the unrefined version [8]. Thus, one of the main purposes of biodiesel refining is to remove all traces of glycerol from the methyl ester product, and to recover the glycerol for further use. At the same time, excess methanol is usually supplied to drive the reaction to economically viable conversion levels. The unreacted methanol in the reactor outlet stream must then be recovered and fed back into the system. The presence of the previously mentioned side reactions complicate these downstream recovery processes, and thus the development of more selective reaction pathways can have indirect benefits on process flow sheets in biodiesel plants.

This study will provide essential baseline information for the potential of heterogeneous catalysts in the commercial production of biodiesel. One of the major biodiesel producers has expressed its interest in the research; the findings of this study can thus provide basis to decide on the feasibility of a long-term process development collaboration with this firm. At the moment, proof of concept is necessary before even considering the eventual commercialisation of the technology. In particular, it is essential to determine whether it is possible to obtain promising yields and whether the process is sufficiently robust to deal with feedstocks of variable quality

3.0 REVIEW OF CaO AS HETEROGENEOUS CATALYSTS FOR TRANSESTERIFICATION

There has been much recent work on the use of various heterogeneous catalysts for transesterification, which are summarised in Table 1. In particular, work on the use of commercial ion exchange resins has given some promising results, with conversions of up to 85% being reported for both pure triglycerides and selected vegetable oils [7, 9-11]. Many of the studies use soybean oil as feedstock; Marchetti [12] reports

the use of waste frying oils, which contain significant amounts of free fatty acids. Conversion levels of about 80% suggest potential for commercial use. Calcium oxide (CaO) catalyst was reported by Zhu *et al.* [13] to give conversion levels of up to 93% using *Jatropha curcas* oil as feedstock. Similar conversion levels were reported for conversion of soya oil [14]. Significantly they also found the presence of water to improve catalyst performance. Repeated use of the catalyst in these tests also showed good durability. Kouzu *et al.* [15] tested different calcium compounds as catalysts for conversion of soybean oil, and found CaO to give the best conversion. Subsequent tests in continuous flow reactors showed some problems with deterioration of catalyst performance with extended use, as well as contamination of the biodiesel product with leached calcium [16]. To date, no studies have been reported on CaO catalysed transesterification of coconut oil.

Table 1: Some Key Findings on CaO Catalysts for Biodiesel Production

Authors	Key Findings
Kouzu <i>et al.</i> [15, 16]	In comparative tests for transesterification of soybean oil using calcium oxide hydroxide and carbonate, CaO was found to give the best conversion. Some catalyst deterioration reported in subsequent tests. Contamination of product by calcium leaching also observed
Liu <i>et al.</i> [14]	Transesterification of soybean oil using CaO catalyst. Maximum conversion of 95% was reported at 300% excess methanol, 8% catalyst dose at $T = 65\text{ }^{\circ}\text{C}$ and 3 h reaction time. Presence of water was found to improve catalyst performance.
Lopez Granados <i>et al.</i> [17]	Transesterification of soybean oil using CaO catalyst. Concluded that CaO is easily deactivated by moisture and CO_2 from the ambient air.
Zhu <i>et al.</i> [13]	Transesterification of <i>Jatropha curcas</i> oil using CaO. Conversion of 93% was reported at $T = 70\text{ }^{\circ}\text{C}$, $t = 2.5\text{ h}$, 200% excess methanol and 1.5% catalyst dose.

4.0 OBJECTIVES

Recent findings described in the previous sections suggest that the use of heterogeneous transesterification catalysts for biodiesel production provides a promising alternative to conventional processes. It is necessary to conduct tests applied specifically to the production of CME, in order to determine the commercial potential of this technology. The objectives of the study are:

- To determine the suitability of calcium oxide for use as heterogeneous catalysts for the production of CME.
- To determine the effect of temperature, excess methanol, catalyst/oil ratio, reaction time on triglyceride conversion.
- To determine the degree of contamination of the biodiesel product with leached calcium.

The study will focus on laboratory scale batchwise processing of coconut oil with methanol using calcium oxide in batch reactions. No detailed theoretical investigation of reaction mechanism or catalyst characteristics will be done; the emphasis was on identifying trends and effects which are useful for future tests on a larger scale.

5.0 METHODOLOGY

Commercially available technical grade calcium oxide was used as catalyst. The catalyst was pretreated using the procedure reported by Zhu *et al.* [13]. The procedure involves immersing the catalyst in ammonium carbonate solution ($C = 0.12\text{ g/ml}$). The catalyst was then filtered, stored in a desiccator for 1 hour, screened (mesh size < 60) and then calcined at $900\text{ }^{\circ}\text{C}$ for 1.5 h. Finally, the catalyst was cooled down to $250\text{ }^{\circ}\text{C}$ and subsequently stored in a desiccator.

Batch tests were conducted with experimental variables at the levels shown in Table 2, based on a 2^4 factorial experimental design [18]. These levels were based in part on the experiments of Zhu *et al.* [13] and also on lessons learned during preliminary tests. Some of the early tests achieved no discernible conversion, as indicated by the complete failure to form a dense glycerol phase. Furthermore, large amounts of catalyst were found to be counterproductive, due to the entrainment of the calcium oxide grains in the glycerol byproduct. These preliminary results were then used to determine the appropriate levels of the experimental variables.

Table 2: Experimental variables and levels

Factor Code	Variable	Low Level (-1)	High Level (+1)
A	Temperature ($^{\circ}\text{C}$)	55	65
B	Time (h)	1.5	3
C	Excess methanol (mole %)	100	200%
D	Catalyst-oil ratio	0.005	0.01

The samples were then put in separatory funnels to allow the biodiesel and glycerol phases to separate. The biodiesel phase was then distilled to remove dissolved methanol, and then analyzed for total glycerol (TG) and calcium content. The TG analysis was performed at the fuel testing laboratory of the Philippine Department of Energy, while calcium analysis was done in-house using atomic absorption spectrophotometry (AAS) after sample digestion with nitric acid.

6.0 RESULTS AND DISCUSSION

The experimental results of the study are summarised in Table 4. The conversion levels shown were computed from the TG analysis. As seen in Equations 1 and 2, the amount of TG in the biodiesel phase decreases as more of the triglyceride (coconut oil) is converted into methyl ester. Based on the typical molecular weight of coconut oil (660 g/mole) the theoretical TG present is 13.9% [7]. On the other hand, pure methyl ester should contain no bound glycerol. Thus, if the biodiesel phase consists mainly of these two components, lower TG values imply higher levels of methyl ester, and thus, higher conversion. Note that, even in the presence of intermediate species (*i.e.*, mono- and diglycerides)

the TG analysis provides a conservative estimate of conversion. The mass fraction of methyl ester in the final product may be computed from the TG analysis using:

$$TG_{\text{sample}} = TG_{\text{CME}} X_{\text{CME}} + TG_{\text{oil}}(1 - X_{\text{CME}}) \quad (3)$$

Where TG_{sample} , TG_{CME} and TG_{oil} are the mass percentages of TG in the sample, in CME and in unconverted coconut oil, respectively. As noted above, TG_{CME} is 13.9% and TG_{oil} is 0%. Furthermore, since the theoretical yield for the transesterification based on Equation 2 is 1 kg of CME for every kg of coconut oil, X_{CME} is also equivalent to conversion in the absence of side reactions. For example, if a batch begins with 1 kg of pure coconut oil, 1 kg of CME will be formed once the reaction reaches 100% conversion. Proportionately less CME will form at lower conversion levels. If, for example, it is found that the $X_{\text{CME}} = 0.9$, then the product must contain 0.9 kg of CME and 0.1 kg of coconut oil; hence, this mass fraction indicates that 90% of the coconut oil initially present has been converted to CME.

The conversion levels computed from the TG analysis of the sixteen batches range from 91.5 – 95.7%. Statistical analysis using the multiple regression toolbox of Microsoft Excel showed

a significant (P -value = 0.025) positive effect of temperature on conversion, while time (P -value = 0.967), excess methanol (P -value = 0.592) and catalyst-oil ratio (P -value = 0.260) were found not to have statistically significant effects for the range of values used in the experiments. Lower P -values indicate more significant influence of a given experimental variable on the response; the threshold for a statistically significant effect is typically 0.05 [18]. By comparison, Zhu *et al.* [13] report 93% conversion of *Jatropha curcas* oil at comparable experimental conditions. The strong influence of temperature on the conversion found in this study contrasts with results reported by Co [7] for ion exchange resin catalysts. The relatively high sensitivity to temperature (as indicated by the statistically significant effect of temperature on conversion) further suggests that the overall reaction rate is determined by the surface reaction rate rather than by mass transfer. This result will have implications for scale-up and design of flow reactor systems, which will differ markedly from those found by Co [7], whose findings suggest a mass transfer controlled reaction mechanism. This difference can be attributed to the smaller particle size of the catalyst used in this study. Furthermore, the ion exchange catalyst used by Co [7] was in supported form, and thus may have exhibited more internal pore resistance.

Table 4: Experimental results

Run	A	B	C	D	TG (%)	Conversion (%)	Ca (ppm)
1	+1	-1	-1	-1	0.96	93.1	72.7
2	+1	-1	-1	+1	0.96	93.1	82.0
3	+1	-1	+1	-1	0.96	93.1	59.6
4	+1	-1	+1	+1	0.99	92.9	54.3
5	+1	+1	-1	-1	1.1	92.1	31.8
6	+1	+1	-1	+1	0.96	93.1	46.4
7	+1	+1	+1	-1	1.1	92.1	57.0
8	+1	+1	+1	+1	0.6	95.7	104.2
9	-1	-1	-1	-1	1.1	92.1	57.5
10	-1	-1	-1	+1	1.11	92.0	81.1
11	-1	-1	+1	-1	1.1	92.1	56.9
12	-1	-1	+1	+1	1.07	92.3	31.0
13	-1	+1	-1	-1	1.09	92.2	135.7
14	-1	+1	-1	+1	1.09	92.2	28.2
15	-1	+1	+1	-1	1.11	92.0	69.9
16	-1	+1	+1	+1	1.18	91.5	33.1

The biodiesel product from all the tests exhibited turbidity. It is unclear at this point if the turbidity is due to the presence of solid calcium oxide particles, droplets of entrained liquid or calcium soap. As previous studies have shown calcium contamination of the biodiesel product, the samples from the study were tested for traces of leached calcium using AAS. The calcium content of the samples was 28.2 – 135.7 ppm, while a blank biodiesel sample (produced using non-calcium based catalyst) was found to contain 42 ppm calcium. Hence, the latter value appears to be the typical calcium level found in the feedstock. The results are also shown in the final column of Table 4. Note that multiple regression analysis showed that none of the experimental factors had a statistically significant effect on the amount of calcium leached into the biodiesel product; all *P*-values calculated were larger than 0.05. Also, the level of trace calcium exhibited only a weak positive correlation ($r = 0.40$) with conversion level. Thus, it is necessary to conduct a more detailed investigation on the response of leaching to various process influences in future studies.

7.0 CONCLUSIONS

This study investigated the effect of temperature, time, excess methanol and catalyst-to-oil ratio on the performance of pretreated calcium oxide catalyst for the transesterification of coconut oil in batch reactions. Performance was measured in terms of conversion, which was calculated from the total glycerol (TG) analysis of the biodiesel phase, and the level of calcium contamination of the product. The tests achieved conversion levels of 91.5 – 95.7%, based on measured TG levels of 0.6 – 1.2%. The specific gravity of the biodiesel phase was also found to be in the range of 0.83 – 0.87, which is also indicative of

high conversion. Statistical analysis of the results of a two-level factorial experimental design shows that only temperature has a significant positive effect on the conversion. The other factors were not found to significantly influence conversion for the range of values investigated here. This statistically significant effect of temperature strongly suggests that the overall reaction rate is controlled by surface reaction kinetics, in contrast to recent work with ion exchange resins that suggests mass transfer controlled reaction rates [7]. The difference may be attributed to the larger particle size and the supported nature of the catalyst used in the previous work. Analysis of the biodiesel samples also showed trace contamination with calcium in the range of 28.2 – 135.7 ppm. This range is similar to the results reported in the literature [15, 16], and is significantly higher than the 42 ppm level measured in a blank sample consisting of biodiesel made using non-calcium based catalyst. However, it is not clear yet whether this contamination is in the form of solid particulates or ionic calcium in entrained liquid. Furthermore, none of the experimental variables investigated showed a statistically significant effect on calcium levels in the biodiesel product. An extension of this work in the future will be the use of catalyst in continuous-flow packed bed reactor experiments. More detailed investigation of the reaction mechanism, as well as catalyst robustness and durability, should also be carried out. Furthermore, it is essential to test the effectiveness of the catalyst on a wider variety of biodiesel feedstocks.

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PROFILES



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