

**THE USE OF HETEROGENEOUS METAL ALKOXIDE
CATALYSTS IN BIODIESEL TRANSESTERIFICATION
REACTIONS**

A Senior Scholars Thesis

by

WILLIAM LLOYD ROONEY, JR.

Submitted to the Honors and Undergraduate Research
Texas A&M University
in partial fulfillment of the requirements for the designation as

UNDERGRADUATE RESEARCH SCHOLAR

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Major: Biological and Agricultural Engineering

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ABSTRACT

The Use of Heterogeneous Metal Alkoxide Catalysts in Biodiesel Transesterification Reactions. (May 2012)

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Reactions that involve two immiscible reactants are challenging to perform due to limitations associated with mass transport. This becomes even more challenging when a catalyst is required to progress the reaction. Biodiesel producing transesterification reaction is an example of such an immiscible system that uses lipids and alcohols. The presently used homogeneous biodiesel catalysts circumvent this issue by acting as mild surfactants that stabilize the reactants that are immiscible in each other. However, heterogeneous catalysts, though easily separable from the reaction medium, are not known to provide these emulsification abilities. Accordingly, there is a need for a solid (heterogeneous) catalyst that will act as an amphiphile (emulsifier) between the two immiscible transesterification reactants. This solid catalyst would stabilize the emulsified the reactants and catalyze the reaction between with its active sites.

Amphiphilic nanoemulsion catalysis is a very new (though not unproven) concept and there remains much work to do to develop optimal conditions, and methods for the preparation of such novel catalysts. In this research, we attempted to attach titanium isopropoxide to solid aluminum hydroxide and determined the effects of method of attachment and other variables upon its use in the transesterification process. FTIR was used to ascertain the attachment of the titanium alkoxide to the alumina substrate by noting the presence or absence of a peak near 1080 cm^{-1} . Three transesterifications were performed on selected experiments and the fatty acid alkyl ester contents of the product mixtures were measured. Results suggest that the procedures tested could produce an economically viable catalyst in the future, though more research is necessary to determine the optimal catalyst composition.

DEDICATION

To my Dad, Dr. Bill Rooney, and Grandfather, Dr. Lloyd Rooney, who gave me my first research experiences and have inspired me to think critically about each action I have taken.

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CHAPTER I

INTRODUCTION

Renewable energy sources will play a significant role in the energy future of the world. With the exponential increase in demand for energy brought about by the exploding economies of the developing world (especially India and China) there will be a tremendous need for new sources of energy dense liquid transportation fuels that are sustainable in the long term.¹ This, combined with the project that world oil production has peaked and is declining or will peak and begin to decline in the near future necessitates the investment of time and research towards developing alternative energy sources.² In recent years there has been an explosion of interest in the area of biofuels.

Biofuels

Biofuels are gaseous, liquid, or solid fuels that are produced from organic materials.¹ Potential production sources for biofuels include grains such as corn, oilseed crops such as soybeans, and lignocellulosic crops such as switchgrass. Biofuels are of interest to energy consumers because of their potential to play a significant role in reducing global conventional fossil fuel use.

This thesis follows the style and format of *Energy and Fuels*.

Biodiesel

Biodiesel is an alternative fuel source that has demonstrated the ability to chemically replace diesel fuel in conventional engines. The American Society for Testing of Materials (ASTM) defines biodiesel as monoalkyl esters of long chain fatty acids derived from a lipid feedstock such as vegetable oil or animal fat.³

Historically the EU has been the leader in biodiesel production, and it continues to produce a large share of the world's annual capacity.⁴ For 2009, worldwide biodiesel production was 17 billion liters annually.⁴ In 2009, the United States produced 2.1 billion liters of biodiesel. This represents a small fraction on the total diesel consumption for the U.S. However, production volume has increased dramatically over the past 10 years and is expected to continue to increase as the price of oil increases in response to increased global demand.^{4,5} Though biodiesel cannot completely replace oil consumption alone there are many advantages of biodiesel that make it probable that it will play a significant role in reducing world oil consumption.

Use of biodiesel in conventional engines

Biodiesel also fits well into the pre-existing transportation infrastructure that the U.S. and other countries have developed after a century of liquid-fueled personal transport. Most, if not all, diesel fueled transportation systems rely on compression ignition engines. Compression ignition engines are similar gasoline engines, but rely on pressure to ignite the fuel-air mixture rather than a spark from a spark plug. Because of their relative simplicity, they may be adapted to many different fuel sources including

biodiesel. Since biodiesel can be used with little to no modification of diesel engine components, compared with other biofuels such as ethanol, its acceptance as an adequate substitute for more conventional fuels is much more likely. Extensive research has already measured the effects, both positive and negative, of replacing diesel with biodiesel in conventional compression ignition engines. When compared to conventionally fueled diesel engines, the specific power output (power produced divided by the weight of the engine) of biodiesel fueled engines was not affected appreciably at below full load conditions, but it was slightly decreased at full load conditions.⁶ Biodiesel also increased the lubricating power of conventional diesel when it is blended with it at any concentration.⁵⁻⁷ However there are tradeoffs to the previously mentioned benefits; for instance, brake specific fuel consumption (BSFC), which measures the amount of fuel per unit power produced, was increased when compared to engines powered by traditional diesel.⁶ Compounding the decrease in BSFC, the energy content of biodiesel is approximately 12.5 % less than its petrochemical counterpart, fuel consumption increased per unit power produced.^{7,8} Additionally, because of biodiesel's higher viscosity and pour point, problems can arise when pumping it into conventional engines at low temperatures. These issues must be addressed if biodiesel is to become an effective substitute for conventional diesel.

Environmental considerations of biodiesel

Biodiesel is a renewable fuel source since the carbon within it comes from the atmosphere in a "real time" basis. It has the benefit of reducing carbon dioxide emissions by up to 78 % over its life cycle when compared to diesel.⁵ Biodiesel is lower

in harmful aromatic hydrocarbons than conventional diesels. Unlike traditional diesel, biodiesel has almost no sulfur present in solution which reduces the harm to catalytic converters of machinery.^{7,8} Particulate matter in emissions, aromatic and polyaromatic compound waste, and carbon monoxide output were positively affected when using biodiesel rather than conventional diesel.^{6,7} Unfortunately, nitric oxide emissions were significantly increased when using biodiesel as a fuel source, presenting another challenge to address before the mainstream use of biodiesel.^{7,9}

Biodiesel production methods

Biodiesel is currently produced from animal fat and vegetable oil feedstocks by the reaction of the contained triglycerides with an alcohol in a process called transesterification. As shown in Figure 1, transesterification is the reaction of a triglyceride source with an alcohol such as ethanol or methanol, to produce monoalkyl fatty esters (biodiesel) and glycerin (a byproduct).⁵

This process was developed in the 1940s as a method for removing glycerol from soap production processes so that it could be used in the production of weapons.⁵ Since then the desired product has changed but the reaction remains highly useful. This process requires the use of a catalyst to facilitate the reaction between the alcohol and the fatty acid.

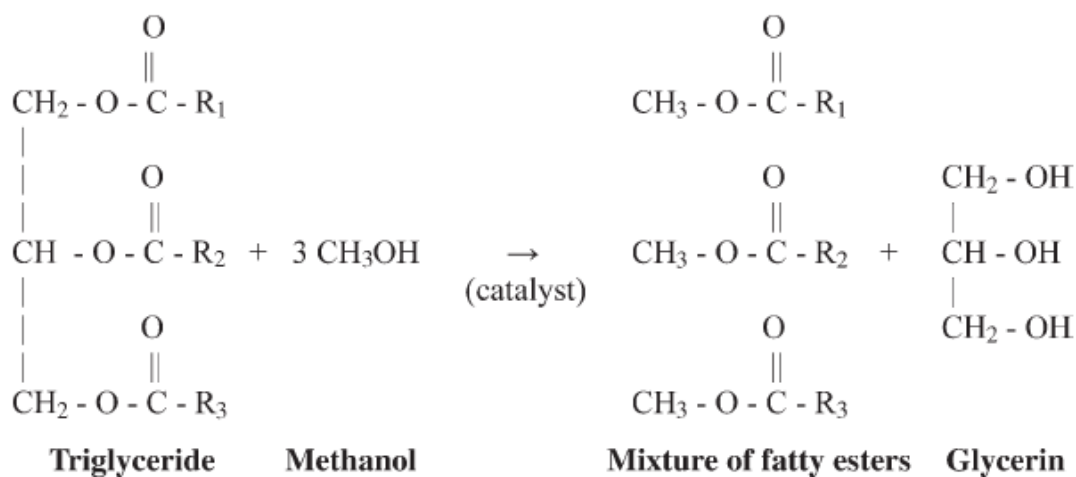


Figure 1. The biodiesel production transesterification reaction. From J. Van Gerpen / Fuel Processing Technology 86 (2005)

Current methods of production

There are two main catalytic routes that have been developed to promote this reaction: homogenous catalysis and heterogeneous catalysis.

Heterogeneous catalysts

A heterogeneous catalyst is not in the same phase as its substrate. There are a great multitude of possible choices for such a catalyst. Some previously studied heterogeneous catalysts include: metal oxides such as $\text{KOH/ZrO}_2\text{-SiO}_2$, $\text{Co}_2\text{O}_3\text{-SiO}_2$, $\text{Mo}_2\text{O}_5\text{-SiO}_2$, $\text{Na}_2\text{O-SiO}_2$, $\text{ZrO}_2\text{-SiO}_2$; metal powders of nickel and palladium; cast iron shavings; steel shavings; zeolites; salts and; ion exchange resins.^{7, 10-14} Current literature presents the opinion that the best performing heterogeneous catalysts are zeolites, specifically those of the ETS-10 group.^{6, 11, 14} Even with the development all these methods the challenge of producing an economically viable product from these catalysts is largely

unmet.¹¹ At present the heterogeneous catalytic systems require tremendous heat and pressure and are fraught with downstream separation problems which make them too costly to be implemented commercially at present.¹⁵

Homogenous catalysts

A homogenous catalyst is in the same phase as the substrate(s) that it works upon. There are two routes that homogenous catalysts can take: alkaline catalysis, and acid catalysis.¹⁶ The acid catalysis route increases the yield of biodiesel because it catalyzes free fatty acid transesterification reactions, whereas alkaline catalysts do not.¹⁰ However, the acidic reaction proceeds at much slower rate than the alkaline catalyzed reaction, so alkaline catalysis is more commonly used.¹¹ The most commonly used base catalysts are the easily available potassium hydroxide and sodium hydroxide.^{3, 5, 8, 11, 15} Basic homogenous catalysts give very high conversion ratios (near 100%) in a relatively short amount of time (generally less than 1.5 hours).^{10-12, 15} The reaction mechanism of base catalyzed transesterification is shown in Figure 2.

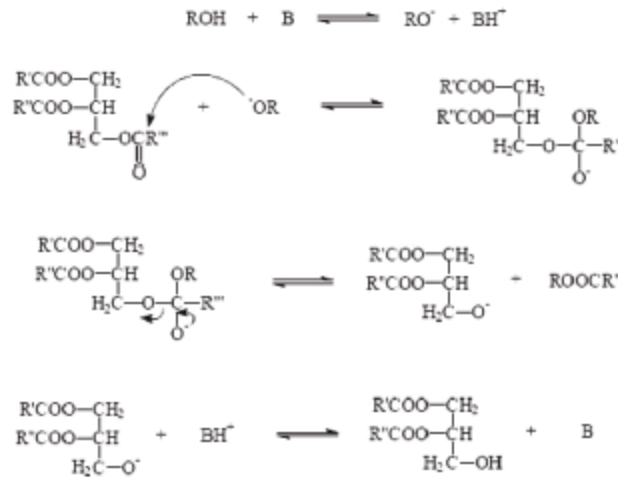


Figure 2. Mechanism of base catalyzed transesterification. From Nawaratna et al, 2010.

Problems with the current production method

There are several problems with the current method of production (homogenous catalysis). First the immiscibility of the alcohol and the oil reactants severely limits the reaction's kinetics (speed) because of the limited interfacial surface area in which the reaction takes place.¹¹ The contact surface area between the two reactants dictates, on a microscopic scale, how quickly a reaction will proceed because it influences the probability that the two reactants will interact with enough energy to react and form the product. The obvious solution to this immiscibility would be to add a liquid surfactant (soap). However, this is not economically feasible because it creates downstream separation problems. Surfactants are soap-like substances that help two immiscible substances to mix by providing an interface between them. Although the widely used alkaline metal hydroxides are known to provide this much needed emulsification, homogeneous catalysts are difficult to separate and add costs to the process.

Possible solutions to this problem

The use of amphiphilic heterogeneous catalysts would both stabilize an emulsion of the non-polar and polar reactants and have active sites to catalyze the desired transesterification. This seems to alleviate the issues discussed above. Heterogeneous catalysts would also alleviate environmental concerns with disposal of waste catalyst as they could be easily reused.¹⁵ Amphiphilic nanoemulsion catalysis is a very new (though not unproven) concept and there remains much work to do to develop the ideal condition, and method for the preparation of such novel catalysts.

The central hypothesis of the work is that the titanium isopropoxide will provide a better alternative (solid) catalyst for the use in biodiesel transesterification reaction. The objective of this research is to develop a method to attach the active titanium isopropoxide catalyst onto a solid support. Then we will evaluate the performance of the hybridized catalyst in soybean oil transesterification to biodiesel.

Evidence that this technique is feasible

There are several preliminary studies which may be found in scientific literature that show positive results for the use of amphiphilic metal alkoxide catalysts. Nawaratna, et al, describe the mechanism by which it would be possible to attach the metal alkoxide to the substrate that is investigated in this research.¹¹

CHAPTER II

METHODS

Overview

Aluminum hydroxide (Sigma Aldrich) was used as the support for attachment and an equal amount (0.5 g) of substrate was used for all experiments. The Aluminum hydroxide was placed in an Aluminum drying dish (VWR International, 57 mm dish with tab) and solutions containing various concentrations of titanium isopropoxide (Aldrich Chemical Company), diluted in isopropyl alcohol (Macron Chemicals), were added to it using the impregnation method. After the final drying was completed, the samples were analyzed using FTIR to determine if attachment took place. The effectiveness of using the experimental products as catalysts in the transesterification reactions was then studied in those experiments that showed IR spectra which suggested attachment had occurred. Three replicates of each experiment were carried out to increase statistical significance.

The impregnation method

Preliminary experiments suggested that a method that slowly impregnates the aluminum hydroxide surface with the titanium isopropoxide through a series of small additions would likely yield the best results. Therefore the impregnation method was used for all samples. This method involved adding the titanium isopropoxide/isopropyl alcohol solution in 1 mL increments to the substrate and allowing the sample to dry completely

in a 98 °C oven between additions. The samples needed approximately 2 hours between additions for drying to occur. This allowed for the slow attachment of the catalyst onto the substrate surface.

Factors tested

Two main factors affects upon catalyst creation were studied: the catalyst to substrate mole ratio, and the dilution rate of the titanium isopropoxide in the isopropanol.

Catalyst to support ratio

This factor is the molar ratio given by (1):

$$\text{Catalyst Ratio} = \frac{\text{Moles of Ti(OCH(CH}_3)_2)_4}{\text{Moles of Al(OH)}_3} \quad (1)$$

The catalyst ratio describes the amount of catalyst loaded per mole of aluminum hydroxide. Three levels of catalyst ratio were tested in this research: 0.5, 1.0, and 1.5.

Dilution factor

Dilution rate describes the composition of the titanium isopropoxide/ isopropyl alcohol solution. We define this factor as the volume percentage of the titanium isopropoxide in the solution. Because the total amount of solution added to the substrate changed the dilution factor essentially describes rate at which the titanium isopropoxide was added to the aluminum hydroxide substrate. Three levels of dilution were used in this experiment: 10%, 30%, and 50%.

Analysis

Fourier Transform Infrared Spectroscopy (FTIR) was used to determine if attachment had occurred in each sample. Those samples that showed the most probability of having the desired attachments were used as catalysts in a transesterification process results of which were analyzed using gas chromatography.

IR analysis

Infrared spectrums of all samples were created using a Nicolet iS10 Thermo Scientific FTIR analyzer. The IR analysis was used to determine whether or not attachment had taken place by comparing the spectra of Alumina, and Titanium dioxide with that of the sample.

Transesterification

The transesterification reaction was done using 100 mL soybean oil and 45 mL of isopropyl alcohol. The catalyst was dissolved in the isopropanol using sonication to increase surface area. The catalyst/isopropanol mixture was then added to the soybean oil heated to 200°C in a continuously mixed reaction pressure thermal reactor (4570-Parr Instrument, Moline, IL, USA, Figure 3) with a maximum operating temperature of 500°C and a maximum pressure of 5000 psi. Once the sample reached 200°C the reaction was monitored by removing samples and recording the pressure every hour for 3 hours. The vessel was then cooled and the contents properly discarded.



Figure 3: The continuously mixed high temperature and high pressure reactor . (4570-Parr Instrument, Moline, IL, USA)

Gas chromatography

The transesterification samples were analyzed using a gas chromatograph (Agilent Technologies 6850) and the results compared to those of conventionally produced biodiesel to determine if the catalyst was efficient.

CHAPTER III

RESULTS

IR spectrographs

Table 1 presents the average IR spectrograph peaks for each experiment and those for the reference samples of Alumina and Titanium dioxide. The three replicates of each experiment had nearly identical IR spectra. Since IR spectra are unique to chemical compounds, the changing spectrographs between samples allowed us to determine if attachment had taken place or not. From Figure 4 we see that the Ti-O-Ti bond vibration is generally near 1120 cm^{-1} and the Al-O-Al peak was found near 1020 cm^{-1} . From these references we infer that the characteristic bond vibration of a Ti-O-Al bond should be in between these two values in the range of 1060 to 1100 cm^{-1} .

Each of the spectrographs from the experiments was analyzed to look for peaks corresponding to Ti-O-Al bonds in the $1060 - 1100\text{ cm}^{-1}$ region. Additionally the peaks corresponding to Al-O-Al, and Ti-O-Ti were recorded if they were present. Table 1 presents the average IR spectrograph peaks for each experiment and what bonds these correspond to in the 1000 - 1140 cm^{-1} wavenumber region. As shown in Figure 5 the three replicates of each experiment had nearly identical IR spectra in all cases.

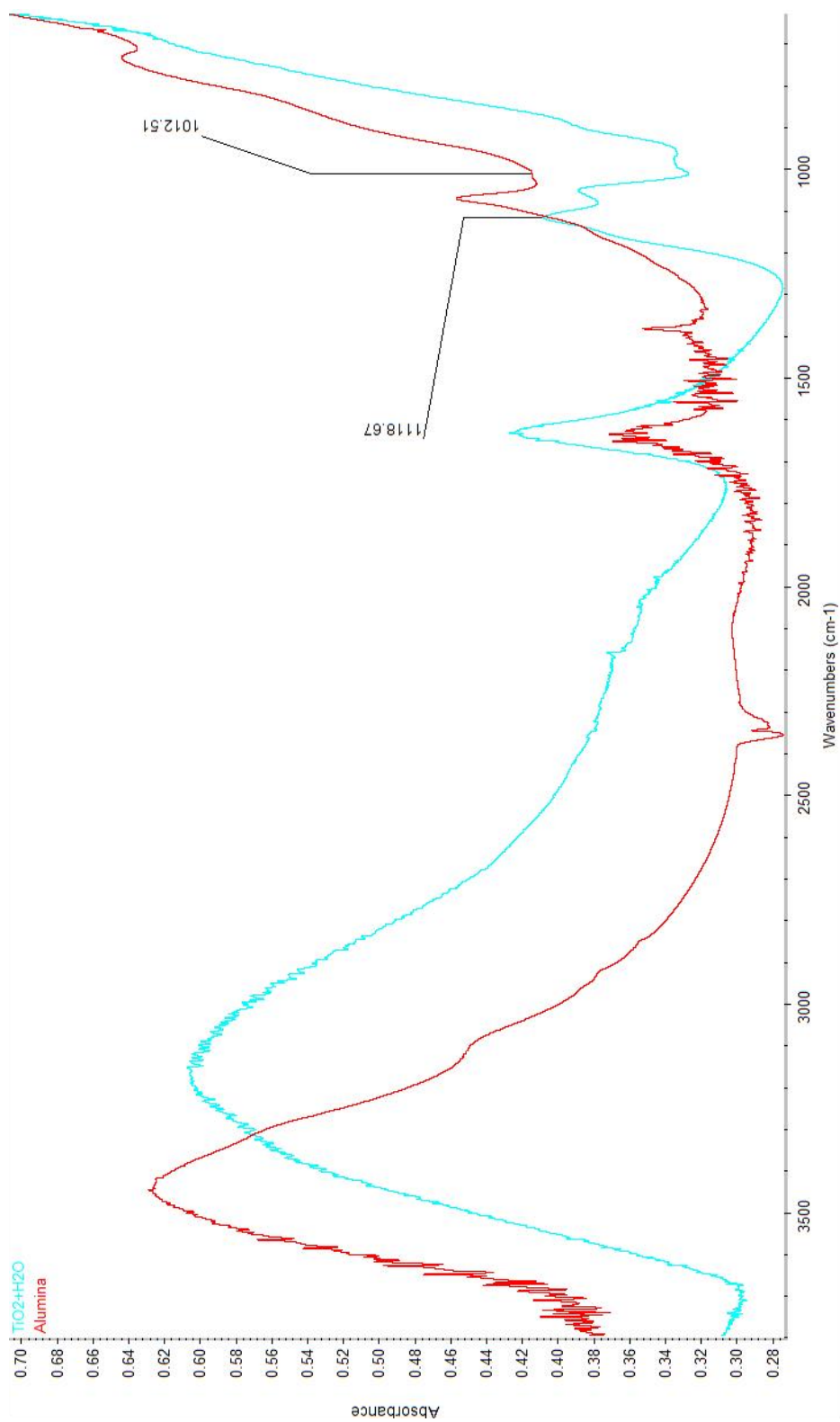


Figure 4: Alumina and Titanium dioxide reference spectra. The peak at 1118 corresponds to the Ti-O bond vibration and the peak at 1012 corresponds to the Al-O bond vibration. From these peaks we infer that the Al-O-Ti bond vibration should be found in between 1060 to 1100 cm⁻¹

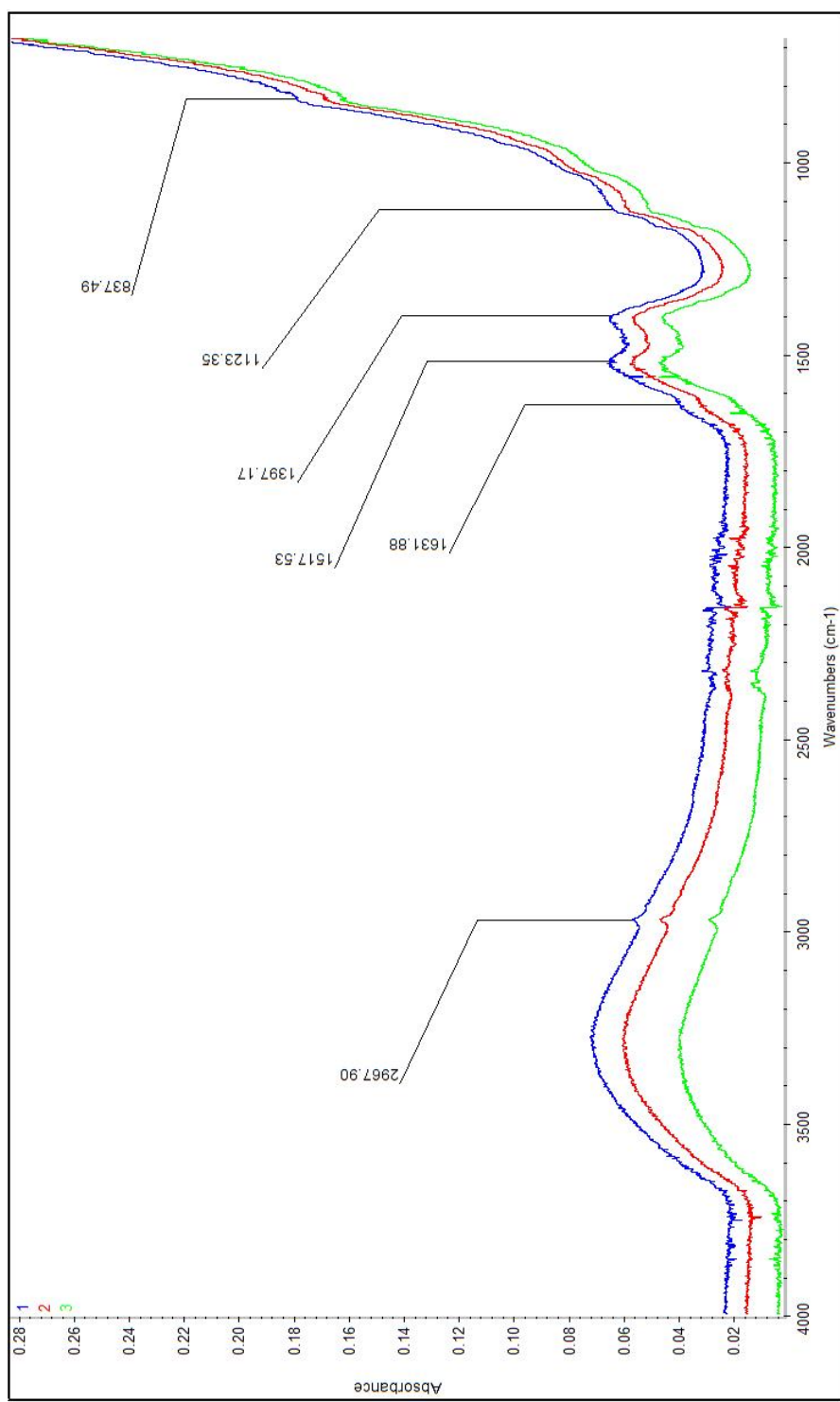


Figure 5: IR spectrograph of the three replicates for Experiment A (Replicates 1, 2, and 3). The consistency among the replicates in this experiment was typical of the other experiments.

From Table 1 we see that the presence of the Al-O-Al bond or the Ti-O-Ti bond seems to preclude the formation of the desired Al-O-Ti bond. From this it was inferred that in experiments A, B, G, H, and I the desired attachment did not occur while in experiments D, E, and F the attachment likely took place. Experiment C was unique in the presence of both an Al-O-Al peak and the Ti-O-Al peak though both were smaller in magnitude than the analogous peaks in the other samples.

Table 1: IR spectrograph peaks

Experiment	Catalyst: substrate ratio	Dilution	Peak Location(s) (cm-1)	Al-O-Al Present?	Ti-O-Ti Present?	Ti-O-Al Present?
Al-O-Al Standard			1012	Yes	No	No
Ti-O-Ti Standard			1118	No	Yes	No
A	0.5	0.1	1021, 1123	Yes	Yes	No
B	0.5	0.3	1018, 1118	Yes	Yes	No
C	0.5	0.5	1021, 1091	Yes	No	Possibly
D	1.0	0.1	1085	No	No	Yes
E	1.0	0.3	1082	No	No	Yes
F	1.0	0.5	1082	No	No	Yes
G	1.5	0.1	1012, 1124	Yes	Yes	No
H	1.5	0.3	1124	Yes	Yes	No
I	1.5	0.5	1018, 1121	Yes	Yes	No

Transesterification Reactions

From these IR spectrographs three experiments, B, F, and I, were chosen to be representative of their catalyst loading ratios in a transesterification reaction process.

Figure 6 presents the biodiesel content of the reaction mixture from 0 – 3 hours of reaction time for each of the samples. The final transesterification yields from the

reactions were all within the expected range of 15-40% biodiesel. Unexpectedly, there was a negative correlation between catalyst: support ratio and the final biodiesel content of the reaction vessel.

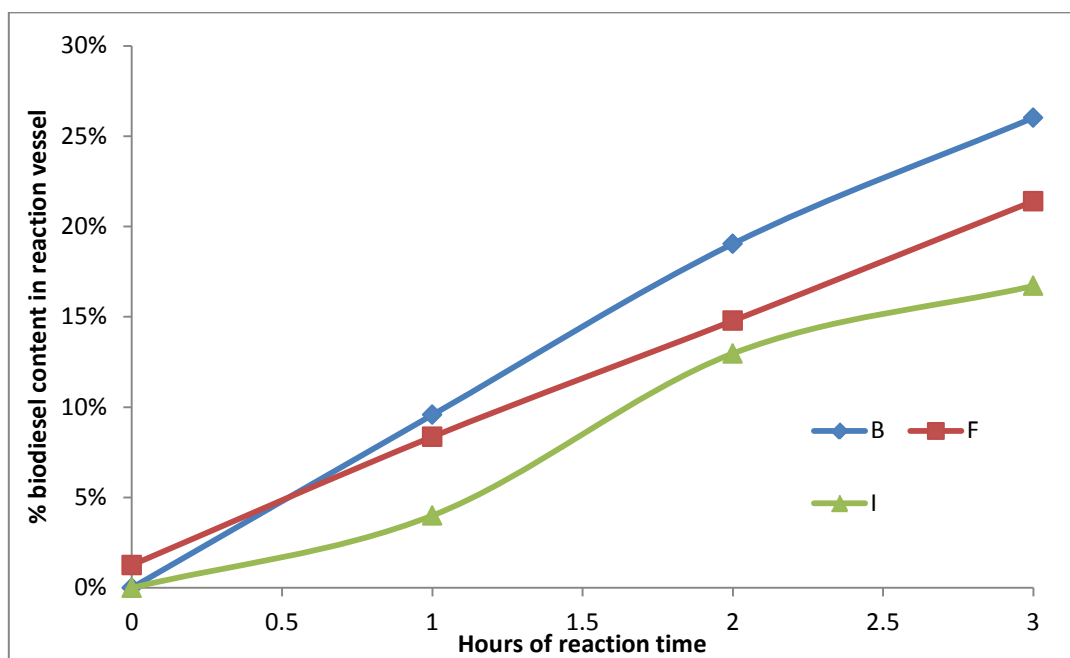


Figure 6: Biodiesel content in transesterification reactions performed. The reaction was run for 3 hours after the reaction vessel reached a temperature of 200°C

CHAPTER IV

SUMMARY AND CONCLUSIONS

In comparing the IR spectra of the newly formed catalysts we found evidence of the attachment of the titanium isopropoxide catalyst to the alumina support. The newly formed amphiphilic heterogeneous catalysts performed adequately in the biodiesel transesterification reaction. These results confirm the possibility of creating novel, amphiphilic, heterogeneous catalysts by an impregnation method, and provide a basis for a larger, more intensive study in this area. While additional research is still necessary to optimize the catalyst development process, a catalyst to substrate ratio of 1:1 or less seems to hold the most promise for future endeavors. The negative correlation between catalyst to support ratio and biodiesel production suggests, at a minimum, that increasing the amount of catalyst per unit of aluminum support, may be ineffective at increasing the catalytic ability of the newly formed catalyst. The reason for this unexpected result is unknown and needs to be investigated more fully to determine factors which contribute to it. A comprehensive study of the impact of the variables tested in this research (dilution rate and catalyst loading ratio), and other variables such as reaction temperature, and the amounts of reagents and catalysts used in the reaction should be undertaken to determine the conditions for optimal conversion. A method of quantifying the increase in production due to the amphiphilic action of the new catalyst should also be developed.

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