

PRODUCTION OF RENEWABLE DIESEL FUEL

Final Report

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Carlo Munoz, Jon Van Gerpen, and Brian He

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16. Abstract Vegetable oils have been investigated as a way to provide a renewable source for diesel fuel. A successful approach to using vegetable oils in diesel engines has been transesterification of the oils with simple alcohols to produce mono-alkyl esters, or <i>biodiesel</i> . A recent development in the area of alternative diesel fuels is a fuel produced from vegetable oils and animal fats using specially modified hydrogenation processes in a conventional petroleum processing facility. This fuel is usually called <i>renewable diesel</i> . This project has focused on developing an understanding of the processes involved with renewable diesel production from a variety of bio-based feedstocks. The project has determined that Raney nickel is an effective catalyst for hydrogenation and deoxygenation of fatty acids, which is a key step in the production of renewable diesel. We have also determined that deoxygenation can take place in a hydrogen-lean environment so that fatty acids can be deoxygenated without having to be completely hydrogenated. This is an important observation because hydrogenation affects the fuel's cold flow properties by increasing its melting point. We are continuing to explore decarboxylation without full hydrogenation as a way to provide renewable diesel with superior low temperature properties.			
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EXECUTIVE SUMMARY

Vegetable oils have been investigated as a way to provide a renewable source for diesel fuel. A successful approach to using vegetable oils in diesel engines has been transesterification of the oils with simple alcohols to produce mono-alkyl esters, or *biodiesel*. A recent development in the area of alternative diesel fuels is a fuel produced from vegetable oils and animal fats using hydrogenation processes in a conventional petroleum processing facility. The processing eliminates the oxygen from the fuel which increases its energy content. It also eliminates the carbon-carbon double bonds, which improves the fuels cetane number and oxidative stability. This fuel is usually called *renewable diesel*.

This project has focused on developing an understanding of the processes involved with renewable diesel production from a variety of bio-based feedstocks. The project has determined that Raney nickel is an effective catalyst for hydrogenation and deoxygenation of fatty acids, which is a key step in the production of renewable diesel. We have also determined that deoxygenation can take place in a hydrogen-lean environment so that fatty acids can be deoxygenated without having to be completely hydrogenated. This is an important observation because hydrogenation affects the fuel's cold flow properties by increasing its melting point. We are also exploring decarboxylation without full hydrogenation as a way to provide renewable diesel with superior low temperature properties.

In addition, substantial conversion of double bonds from the cis configuration to the trans configuration has been observed during the early stages of hydrogenation and this may be an effective way to optimize the trade-off between oxidative stability, cetane number, and cold flow properties.

DESCRIPTION OF PROBLEM

The term *renewable diesel* is generally used very broadly to designate fuels other than alkyl esters that are produced from vegetable oils and animal fats. Because there are currently no specifications for these fuels, there is considerable variation in how the term is used. One class of renewable diesel fuels are those that are produced by bringing oils and fats into a petroleum refinery, removing oxygen, adding hydrogen and producing a fuel rich in normal alkanes. The fuel may be co-processed with a conventional petroleum stream or may be processed separately and maintained as an identifiable stream (Rantanen et al. 2005). Normal-alkanes (*n*-alkanes) are prominent constituents of conventional diesel fuel, so if the renewable diesel is blended with conventional diesel fuel, it may be difficult to distinguish it from the fossil fuel.

Over 20 years ago, the CANMET Energy Technology Centre in Ottawa, Canada, developed technology for hydrotreating vegetable oils, animal fats, wood oils, and tall oil (a by-product of paper production) to produce a high-cetane diesel fuel additive (Monnier et al. 1998). Their product, known as SuperCetane, consisted mostly of *n*-alkanes and had a cetane number of around 100. The process never became popular because of the high price of oils and fats compared with fossil-based crude oils. Similar processes are now proposed to use conventional petroleum refining processes such as hydrogenation, cracking, and isomerization to convert oils and fats to gasoline, aviation fuel, and diesel fuel.

Because the oils and fats undergo extensive chemical modification, the actual fatty acid profile does not determine the properties of the final product as is the case for biodiesel. The primary concern is for metallic contaminants such as phosphorus that can poison catalysts. Oils and fats for hydrogenation processes should be degummed to remove the natural phospholipids that are found in these feedstocks.

Animal fats are frequently found to contain sulfur from protein carry-over during the rendering process. Vegetable oils from oilseeds with high glucosinolate content can also contain sulfur. However, since hydrogenation is frequently used for desulfurization, these contaminants should not present a problem.

Hydrotreating is a common process in petroleum refineries and is normally used to remove sulfur, nitrogen, and metals from petroleum-based feed streams such as heavy gas oil or vacuum gas oil. It has been known for some time that fatty acid-based materials can be converted to straight chain alkanes by hydrotreating.

Researchers have identified the reaction pathways that are relevant to the conversion of oils and fats to hydrocarbons (Huber et al. 2007). The first step in the process consists of hydrogenation of the C=C bonds, as shown in Figure 1. The C=C bonds are located in the fatty acid chains designated by R₁, R₂, and R₃. The number of chains with C=C bonds depends on the level of unsaturation in the fatty acid profile. Over 85% of the fatty acid chains in soybean oil contain one or more C=C bonds. More saturated feedstocks such as animal fats and palm oil contain 55–60% unsaturated fatty acids.

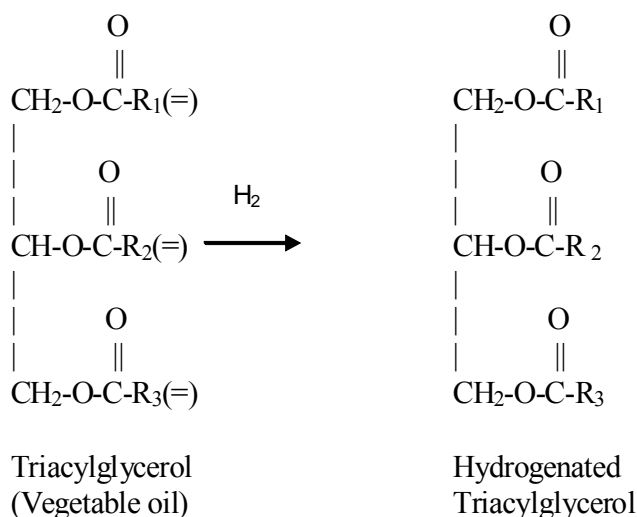


Figure 1: Hydrogenation of olefinic (C=C) bonds.

The hydrogenated triacylglycerols (triglycerides) are further reacted to split the fatty acid chains from the glycerin backbone as shown in the first step of Figure 2. The glycerin will go to propane or lighter gases. The saturated fatty acid chains undergo dehydration, decarbonylation, or decarboxylation reactions to produce normal alkanes between 15 and 18

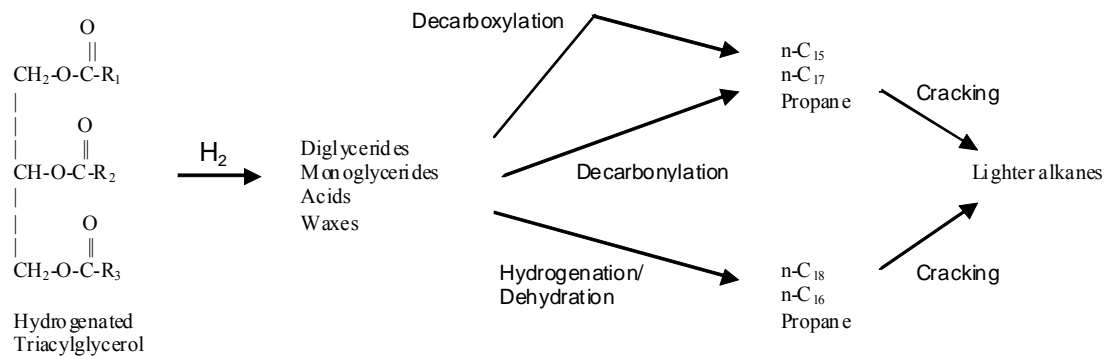


Figure 2: Pathways for conversion of hydrogenated triglycerides to alkanes (Huber et al. 2007).

carbons long. These long chain *n*-alkanes have excellent cetane numbers but also have cold flow properties that are even worse than the alkyl esters in biodiesel. The *n*-alkanes will either be cracked to produce shorter *n*-alkanes, or isomerized to branched alkanes depending on the anticipated final product.

The global reaction for the hydrogenation of a triglyceride such as triolein would be:



which shows that 6 moles of hydrogen are required for each mole of triolein. This reaction should be taken as a minimum since a portion of the oxygen will also be released as water thus requiring additional hydrogen. The reaction requires at least a molecule of hydrogen for each double bond plus another molecule to terminate the radical ends created by the decarboxylation. Triolein can be taken as characteristic of a vegetable oil like soybean oil although a more saturated oil like beef tallow would have somewhat less hydrogen requirement.

Hydrogen tends to be in short supply in refineries. The source of this hydrogen is likely to be reforming of methane and other low value short-chain hydrocarbons, possibly including the

propane produced in the reaction. In any case, the cost of the hydrogen will be an important factor in the economic viability of this technology.

The normal alkanes provide a good cetane number and will be compatible with existing engines and fuel distribution infrastructure. Because the oxygen that was originally in the oil or fat has been removed, the energy content of the renewable diesel fuel will be higher than the original vegetable oil or biodiesel. However, alkanes tend to have low densities so the energy content of a gallon of renewable diesel will still be slightly less than conventional diesel fuel, which may contain 30 to 35% high-density aromatic hydrocarbons. This difference is small enough that it is unlikely consumers will see any mileage penalty from using the fuel. One additional caution for the fuel is that *n*-alkanes tend to have poor lubricity and poor cold flow properties. This can usually be managed with additives.

If a gasoline-grade product is desired, the alkanes may also be isomerized to provide the chain branching needed to raise the octane number to a level that will be suitable for spark-ignited engines. Depending on the extent of processing of the long-chain alkanes and the diversity of products produced, a fractionation process might be needed to ensure the quality of the final product.

Our primary objective is to develop the process technology needed to convert animal fats and vegetable oils to renewable diesel. We want to be able to conduct economic analyses and to optimize the properties of the fuel that is produced. An additional objective is to determine whether the production technology for these compounds, which has mainly been focused on large petroleum refining facilities, can be implemented on a smaller scale. If this can be done, existing biodiesel production facilities could retrofit their plants with this technology. The fuel is expected to be easier to integrate into the existing fuel infrastructure than biodiesel.

APPROACH AND METHODOLOGY

Hydrogenation is the main process that has traditionally been used to make renewable diesel. It is also widely used in the fats and oils industry to upgrade highly unsaturated oils. The hydrogenation of vegetable oils is a chemical process by which hydrogen is added to a carbon-carbon double bond in the glycerides of an oil molecule. The reaction takes place in the presence of a catalyst, usually nickel, and the product is an oil of higher consistency, stability and oxidation resistance.

Industrially, the process is carried out in tanks with conventional blade stirrers, hydrogen bubbling and the catalyst particles in suspension, usually working at 150°C – 220°C with pressures between 69 kPa (10 psi) and 413 kPa (60 psi), using a Raney nickel catalyst. Other alternative catalysts are palladium, platinum and gold supported on alumina. These catalysts have a higher activity, which allows the hydrogenation reaction to take place at lower temperatures (80°C – 120°C).

In the experiments conducted here, the oil and catalyst will be contained in a constant volume reactor. The hydrogen will be added from an external gas cylinder with an initial starting charge as indicated by the initial pressure of hydrogen. The reaction will be initiated by gradually heating the contents of the reactor.

In renewable diesel production, the first step is to hydrogenate all the fatty acids. Hydrogenating the fatty acids increases the fuel's heating value and will also modify some properties like cold flow and oxidation stability. As the degree of saturation increases, the oil's viscosity and melting point increase. When a fatty acid is saturated with hydrogen, all the fatty acid's double bonds are eliminated by the addition of hydrogen atoms to those double bonded carbon atoms.

The difference between the melting points of the fatty acids can be significant. The more saturated they are, the higher the melting point. So, while hydrogenation improves some properties (energy content, cetane number, oxidative stability) it worsens others (cold flow). This trade-off is one of the critical issues in the production of renewable diesel.

Hydrogenated vegetable oil should have a higher melting point than its methyl ester. Therefore, for the first experiments, methyl esters were used instead of neat triglycerides (TG).

FINDINGS; CONCLUSIONS; RECOMMENDATIONS

The main objective of the first experiments was to test Raney nickel's ability to hydrogenate and deoxygenate methyl esters. Therefore, the attention was focused on measuring the degree of deoxygenation of the methyl esters, not the degree of hydrogenation. Initially, canola, coconut, and waste vegetable oil were used as feedstocks, and for the first runs a low initial hydrogen pressure (hydrogen lean) of around 300 to 400 psi was utilized.

In these early experiments it was noticed there was a drastic pressure drop at around 75°C, when methyl esters were used as feedstock. This pressure drop was observed in every experiment regardless of the feedstock, indicating a reaction was taking place.

In the first experiment, canola methyl ester was used and the pressure began to decrease at a rate of 21.67 psi per minute when the temperature was around 75°C, before that point, the pressure drop rate was only 1.18 psi per minute. It was later confirmed that the hydrogenation reaction takes place between temperatures of 75°C and 150°C. Also, the average temperature rise rate was 4.88°C per min before the biodiesel reached 75°C. During the 10 minute period when the pressure drop is drastic (hydrogenation), the temperature rise rate was 9.75°C per min, almost twice as fast as during regular heating, indicating an exothermic reaction.

When triglycerides were used as feedstock, the hydrogenation reaction takes place between temperatures of 130°C and 200°C. The pressure rise rate during heating was 8.53°C per min, and during hydrogenation the temperature rise rate was 6.1°C per min. The pressure rise during heating was 6.71 psi per minute but the pressure drop rate during hydrogenation was 51.03 psi per min. The starting hydrogen pressure was 612 psi compared to the 300 psi used for the methyl esters, so that more hydrogen was available and therefore the hydrogenation reaction could be more complete.

Based on these results it was determined that hydrogenation of the methyl esters took place at lower temperatures than triglycerides. Also, the hydrogen was consumed in around 10 minutes, regardless of the feedstock.

Samples of the hydrotreated oil and methyl esters were sent to a local analytical lab in order to determine the composition and concentration of the compounds present in the wax produced by the hydrotreatment. When the samples were analyzed and the results came back, it was noticed that although there were n-alkanes (paraffins) produced, there were also n-alkenes (olefins) and unsaturated free fatty acids present, indicating the presence of unsaturated compounds.

Table 1 shows an example of the product composition of the hydrotreated methyl ester. From these experiments some conclusions can be drawn:

- Raney Nickel is an effective catalyst for hydrogenation and deoxygenation of fatty acids.
- Deoxygenation could take place in a hydrogen lean environment.
- Fatty acids can be deoxygenated without having to be completely hydrogenated.

Hydrogenation affects the fuel's cold flow properties by increasing its melting point.

Hydrotreated methyl esters have lower melting points than hydrotreated triacylglycerides.

This is possibly due to the fact that methyl esters have lower melting points than free fatty acids.

Table 1: Composition of Hydrotreated Methyl Ester

Hydrocarbon	ppm	% w/w	Formula
Pentdecane	1,371.49	2.01%	C ₁₅ H ₃₀
<i>Heptadecene</i>	<i>1,218.05</i>	<i>1.78%</i>	<i>C₁₇H₃₄</i>
Heptadecane	2,812.37	4.12%	C ₁₇ H ₃₆
Hexadecanoic Acid	5,147.60	7.54%	C ₁₆ H ₃₂ O ₂
<i>Octadecenoic Acid</i>	<i>637.96</i>	<i>0.93%</i>	<i>C₁₈H₃₄O₂</i>
Octadecanoic Acid	522.37	0.77%	C ₁₈ H ₃₆ O ₂
<i>Erucic Acid</i>	<i>4,795.52</i>	<i>7.03%</i>	<i>C₂₂H₄₂O₂</i>
Hexadecanoic Acid ME	5,687.70	8.33%	C ₁₇ H ₃₄ O ₂
Heptadecanoic Acid ME	1,310.40	1.92%	C ₁₈ H ₃₆ O ₂
<i>Octadecenoic Acid ME</i>	<i>19,011.18</i>	<i>27.85%</i>	<i>C₁₉H₃₆O₂</i>
Octadecanoic Acid	16,842.12	24.67%	C ₁₈ H ₃₆ O ₂
<i>Eicosenoic Acid ME</i>	<i>2,058.92</i>	<i>3.02%</i>	<i>C₂₁H₄₀O₂</i>
Eicosanoic Acid ME	1,761.71	2.58%	C ₂₁ H ₄₂ O ₂
Docosanoic Acid ME	2,339.78	3.43%	C ₂₃ H ₄₆ O ₂
Unknown	2,742.00	4.02%	-

After the first set of experiments and with confidence that Raney nickel was an appropriate catalyst, it was decided to select a uniform feedstock in order to monitor the conversion of fatty acids into paraffins. Four gallons of refined, bleached and deodorized (RBD) soybean oil were purchased from a local supermarket. The oil was heated and mixed for 30 minutes in order to ensure a uniform blend.

A second series of tests were performed, but this time the main objective was to ensure complete hydrogenation of the oil and the methyl esters. The first step was to determine the fatty acid profile of the mixture of soybean oils. Two independent laboratories analyzed a sample of the soybean oil. The results are shown in Table 2. There was a surprising amount of disagreement between the laboratories. Because the data collected by the UI laboratory is closer to the values from the literature, it was assumed to be correct.

For the first hydrogenation experiment, 100 gr (0.1431 mol) of soybean oil and 5 gr of Raney nickel catalyst were reacted with 0.589 moles of H₂ for 70 minutes at a maximum temperature of 200°C.

Table 2: Soybean Oil Fatty Acid Profile

Fatty Acid	Hydrocarbon	Literature	U of I	Anatek
			% w/w	% w/w
Capric Acid (10:0)	Decanoic Acid	0.00	0.00%	0.00%
Lauric Acid (12:0)	Dodecanoic Acid	0.00	0.00%	0.00%
Mirystic Acid (14:0)	Tetradecanoic Acid	0.00	0.00%	0.00%
Palmitic Acid (16:0)	Hexadecanoic Acid ME	8.00%	10.40%	26.81%
Stearic Acid (18:0)	Octadecanoic Acid	4.00%	4.60%	0.00%
Oleic Acid (18:1)	Octadecenoic Acid	25.00%	24.60%	6.92%
Linoleic Acid (18:2)	Octadecadienoic Acid	55.00%	52.30%	57.92%
Linolenic Acid (18:3)	Octadecatrienoic Acid	8.00%	6.40%	0.00%
Arachidic Acid (20:0)	Eicosanoic Acid	0.00	0.40%	3.52%
Gondoic Acid (20:1)	Eicosenoic Acid	0.00	0.20%	0.00%
Behenic Acid (22:0)	Docosanoic Acid	0.00%	0.40%	4.84%

It was calculated that 0.170 moles of hydrogen were needed to hydrogenate all the unsaturated fatty acids into stearic acid. Thus, 3.46 times the stoichiometric amount of H₂ was used.

Four samples were taken from the experiment and given to the UI Plant Science department for their analysis (contact was Jim Davis). The tests were conducted to investigate any changes in the fatty acid profile with time. The results are shown in Table 3. Hydrogen was added when the pressure was lower than 60 psi, and the initial sample was taken before adding the hydrogen.

Table 3: Fatty Acid Profiles of Vegetable Oil Samples

Fatty Acid	Reference	Raw Soy Oil	1st Sample	2nd Sample	Final Sample
Palmitic Acid (16:0)	10.40%	9.00%	9.10%	9.40%	10.30%
Stearic Acid (18:0)	4.60%	4.00%	24.10%	78.00%	88.50%
Oleic Acid (18:1)	24.60%	43.30%	31.80%	4.80%	-
Trans Oleic Acid (18:1)	-	-	11.90%	2.30%	-
Linoleic Acid (18:2)	52.30%	32.90%	10.50%	32.90%	-
Trans Linoleic Acid (18:2)	-	-	7.40%	-	-
Linolenic Acid (18:3)	6.40%	6.00%	0.40%	6.00%	-
Trans Linolenic Acid (18:3)	-	-	1.60%	-	-
Arachidic Acid (20:0)	0.40%	0.50%	0.80%	1.20%	0.50%
Gondoic Acid (20:1)	0.20%	0.70%	0.50%	-	-
Behenic Acid (22:0)	0.40%	0.40%	0.40%	0.40%	0.40%

Hydrogen was added in two steps, and at 34 minutes after the hydrogenation started, the pressure became constant at 200 psi, indicating that the reaction had reached equilibrium. The heating rate was 8.53°C per min, but during hydrogenation the temperature rise rate had increased to 13.33°C per minute. Hydrogenation is an exothermic reaction and the increase in the heating rate confirms that. On the other hand, during the hydrogenation, the pressure drop rate was 51.03 psi per minute, while during the heating phase it was only 6.71 psi per minute.

One observation that can be made from Table 3 is that large amounts of trans fatty acids are being made early in the hydrogenation process. In nature, virtually all double bonds are in the cis form but hydrogenation is known to produce trans fatty acids. Even though they are unsaturated, trans fatty acids behave more like saturated fatty acids and have poor cold flow properties.

Based on these results, the following mechanism is proposed for the hydrogenation of the fatty acids and double bonds with Raney nickel as catalyst:

1. Cis – trans isomerization of unsaturated fatty acids into their trans isomer.
2. Saturation of the trans isomer into the corresponding saturated fatty acid.



Figure 3: Hydrogenation of soybean oil.

In the photograph shown in Figure 3, it can be seen how the saturated soybean BD (left) is semi-solid at room temperature compared to the untreated soybean BD (right). Also the color of the hydrogenated BD is much lighter than the unsaturated biodiesel.

A similar experiment was carried out with soybean biodiesel as the feedstock instead of the soybean oil. The objective of the experiment was to see if changing the form of the feedstock has an effect on hydrogenation. To investigate this, 100 gr (0.3292 mol) of soybean biodiesel and 5 gr of Raney nickel catalyst were reacted with 0.4640 moles of H_2 for 70 minutes at a maximum temperature of $200^\circ C$.

No samples could be taken from this experiment because no product emerged from the sample valve, therefore the only sample that was analyzed was the final sample recovered from the reactor after the test.

Hydrogen was added in one step, and equilibrium was reached 52 minutes after hydrogenation started. But 40 minutes after hydrogenation, the rate of pressure drop was very slow.

During hydrogenation, the temperature went up and down. It wasn't constantly going up like the last experiment, and this behavior continued until equilibrium was reached. On the other hand, during the hydrogenation, the pressure drop rate was consistent at 51.31 psi per minute, while during the heating phase it was only 10.55 psi per minute.

The final product from starting with the methyl ester had a lower melting point ($\approx 37^{\circ}\text{C}$), whereas the saturated oil had a melting point of $\approx 75^{\circ}\text{C}$. The results of this test are shown in Table 4.

Table 4: Gas Chromatograph Results for Soybean Oil Samples

Fatty Acid	Reference	Raw Soy BD	Final Sample
Palmitic Acid (16:0)	10.40%	9.10%	10.20%
Stearic Acid (18:0)	4.60%	3.90%	88.30%
Oleic Acid (18:1)	24.60%	43.40%	-
Trans Oleic Acid (18:1)	-	-	-
Linoleic Acid (18:2)	52.30%	32.80%	-
Trans Linoleic Acid (18:2)	-	-	-
Linolenic Acid (18:3)	6.40%	6.00%	-
Trans Linolenic Acid (18:3)	-	-	-
Arachidic Acid (20:0)	0.40%	0.50%	0.50%
Gondoic Acid (20:1)	0.20%	0.70%	-
Behenic Acid (22:0)	0.40%	0.30%	0.40%

The production of trans fatty acids has been noted by other researchers and is shown by the graph in Figure 4 extracted from a paper by Koseoglu and Diosady (1984). This graph, shows how trans isomers are formed by hydrogenation with nickel and MR-Cr(CO)_3

catalysts, with nickel being the fastest. It can be seen that in a period of less than 20 minutes both the iodine value drops dramatically (inset graph) and in less than 15 minutes the trans isomers are created, supporting the findings presented here.

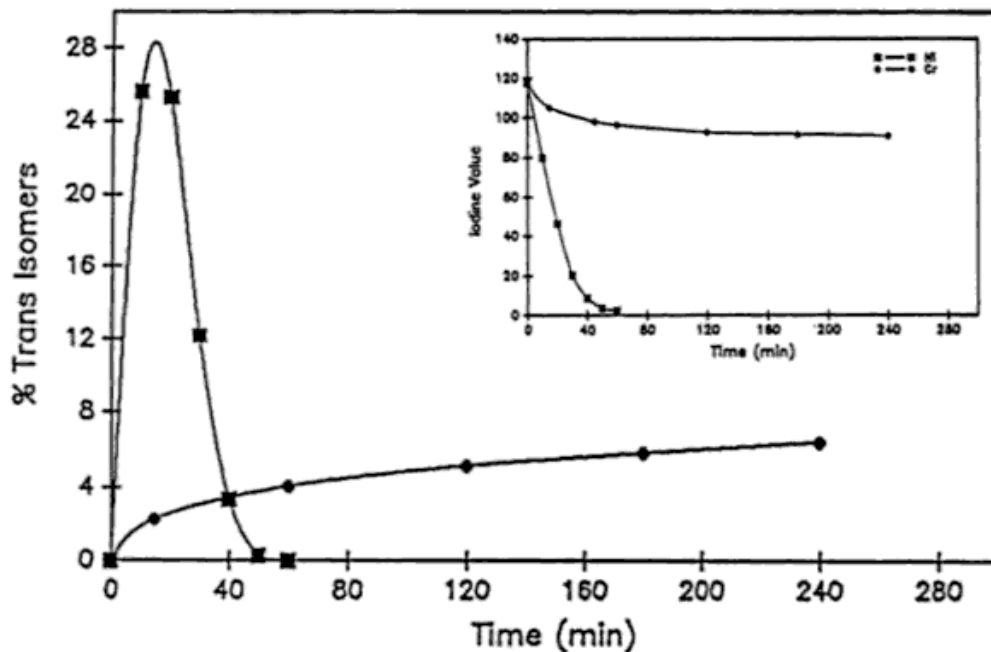


Figure 4: Production of trans fatty acids (Koseoglu and Diosady, 1984).

Adapted from: Koseoglu and Diosady et. al (1984), taken from the Book “Canola and Rapeseed: Production, Chemistry, Nutrition and Processing Technology, F. Shahidi.

Conclusions

The following conclusions were drawn from the research conducted in this project.

- Complete hydrogenation of both soybean oil and soybean methyl esters was achieved in around 40 minutes.
- Cis-trans isomerization affects the fuel’s cold flow properties, it takes place before the hydrogenation, and it could be used to increase the fuel’s cetane number. More tests are required to prove this point.
- Raney nickel is an effective catalyst for hydrogenation.

- For 100 gr of fatty acids, 1000 psi of hydrogen in the batch reactor is more than enough to have complete hydrogenation.

Aspects to Consider for Future Research

- Determine the benefits or drawbacks of cis – trans isomerization
- Explore dehydrogenation as an alternative for improving fuel's cold flow properties.

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