

**FEASIBILITY ANALYSIS OF STEAM REFORMING OF
BIODIESEL BY-PRODUCT GLYCEROL TO MAKE HYDROGEN**

A Senior Scholars Thesis

by

MANOJ JOSHI

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

UNDERGRADUATE RESEARCH SCHOLAR

April 2009

Major: Chemical Engineering

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Approved by:

Research Advisor:

Associate Dean for Undergraduate Research:

Mahmoud M. El-Halwagi

Robert C. Webb

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ABSTRACT

Feasibility Analysis of Steam Reforming of Biodiesel by-product Glycerol to Make Hydrogen. (April 2009)

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Crude glycerol is the major byproduct from biodiesel industry. In general, for every 100 pounds of biodiesel produced, approximately 10 pounds of crude glycerol are produced as a by-product. As the biodiesel industry rapidly expands in the U.S., the market is being flooded with this low quality waste glycerol. Due to its high impurities, it is expensive to purify and use in food, pharmaceutical, and cosmetics industries. Biodiesel producers should seek an alternative method which is economically and environmentally friendly.

This research contains reforming process to covert waste glycerol from a biodiesel industry into sellable hydrogen. This process consists of 850°C reformer, 350°C and 210°C shift reactors for water gas shift reaction, flash tanks, and a separator. It is considered to be the least expensive method.

At 850°C and 1 atm pressure, glycerol reacts with superheated steam to produce gaseous mixture of hydrogen, carbon dioxide, carbon monoxide, and methane. Reformer is a batch process where only 68% of waste glycerol is converted into gaseous mixture. The excess glycerol is recycled back as a feedstock. Water gas shift (WGS) reaction, further convert carbon monoxide into hydrogen and carbon dioxide which is further subjected to separation process to isolate hydrogen from CO₂ and any other impurities. The final product stream consists of 68% of hydrogen, and 27% of CO₂ based on molar flow rate.

DEDICATION

To my father M.K. Joshi, mother S. Joshi and brother A. Joshi.

ACKNOWLEDGMENTS

My first and foremost thanks goes to my research advisor Dr. Mahmoud M. El-Halwagi, Professor and Holder of the McFerrin Professorship at Artie McFerrin Department of Chemical Engineering, Texas A&M University. It has been a great honor and pleasure to be his Undergraduate Scholar. I appreciate all his valuable contributions of time, ideas, patience, and encouragement. Without him, this thesis would not have been possible. I thank all the committee members, academic advisors and professors: Dr. Robert C. Webb, Dr. Lale Yurttas, Dr. John Baldwin, and Dr. Suma Datta for their feedback and support. I would also like to thank the members of Process Integration and Optimization Group - Texas A&M University, Viet Pham - Ph.D. Candidate, and Steven Matthew Chiv - University of Texas for their contributions.

I gratefully acknowledge The Dwight Look College of Engineering and Undergraduate Student Research Grant (USRG) for funding my research.

Lastly, I would like to thank my family for always supporting me through all these years, and for their love and encouragement.

NOMENCLATURE

C/S	Steam / Carbon
WGS	Water Gas Shift
P	Pressure
T	Temperature
t	Time
atm	Atmosphere

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CHAPTER I

INTRODUCTION

The major by-product of biodiesel production, glycerol has become one of the major concerns of biodiesel industries. During the manufacture of biodiesel via transesterification of oils, 100 kg of crude glycerol is produced for every 1 tone of biodiesel, (see figure 1) (Bio-fuels and Glycerol). Crude glycerol from biodiesel contains high percentage of impurities and hence has a low economic value. It consists of 40.4% fatty matter, 33.1% Glycerol, 23.3% methanol, and 3.2% water and ash (Slinn, 2008).

The increase in the biodiesel production in global has increased the production of by-product glycerol and made the market saturated. Glycerol production in the United States already averages more than 350,000 tons per year and in Europe its production has tripled within the last ten years (Bio-fuels and Glycerol).

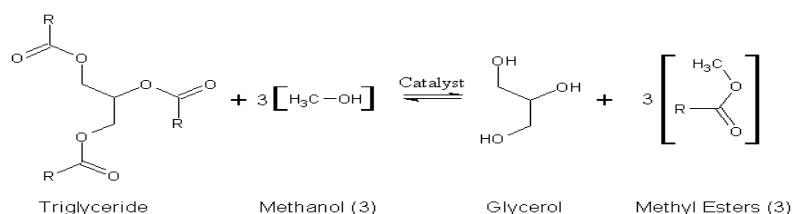


Fig.1. Transesterification of fatty oil.

This thesis follows the style of Biosource Technology.

Pure glycerol has a wide application. But the by-product glycerol is highly impure and cost more capital to purify it. The by-product glycerol can be purified by distillation process and can be use in food and pharmaceutical processing industries. But it's an expensive process to separate the high impurities such as methanol, ash, and fatty matter. It might also be useful to burn as a fuel if blended with fuel oil but it will have a serious environmental threat. These days by-product is sent to water treatment for digestion but it is a slow and expensive process (Slinn, 2008). On the other hand, processing industries couldn't use all the glycerol produced from the biodiesel industries. Hence, finding the valuable alternative to use crude glycerol that is economically and environmentally friendly and that could potentially take all the waste glycerol would be important for the success of the biodiesel industries.

The goal of the research is to analyze the feasibility of steam reforming process of highly impure by-product glycerol to convert into sellable hydrogen. This research also focuses on the further purification of hydrogen from steam reforming process via water gas shift (WGS) reaction. An experiment has been conducted at University of Birmingham, United Kingdom where pure glycerol has been heated at different temperatures to study the effect of the temperature on the conversion of the glycerol into Syn gases. Based on this experimental data, simulation was done on Aspen Plus simulator as shown in design approach section of the report. RK-Soave thermodynamic properties were used in the simulation. The process design consists of reformer (reactor) at very high temperature to carry out the reactions that converts glycerol into hydrogen followed by another reactor

to carry out WGS reaction as shown in the Flowsheet. Further, the techniques of mass integration and heat integration were applied to the process to minimize the total capital investment. The “Process Integration” written by Dr. Mahamoud M. El-Halwagi was used to understand the process integration techniques. The design approach and the flow sheet of the process explain in detail about the process design, calculations, and integration techniques (El-Halwagi, 2006).

Literature review

In recent years, the U.S. has been experiencing historic highs in oil prices. This made researchers and industries to consider the alternative fuel source. Biodiesel is one alternative fuel source everyone is interested with a common interest to reduce the fuel price and to be less dependable on foreign oil. Due to this facts biodiesel production has increased from 0.5 million gallons in 1999 to 145 millions gallons in 2007 as shown in figure 2.

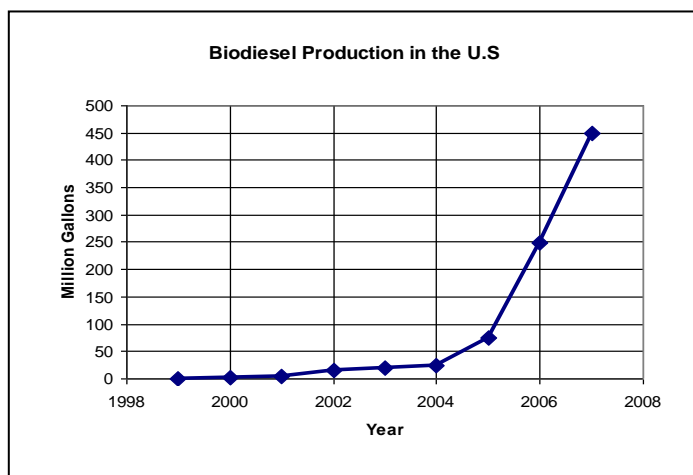


Fig.2. Biodiesel production in the U.S (National Biodiesel Board, 2008).

As a result by-product waste glycerol is produced in large amount in the ratio of 10 kg for every 100 kg of biodiesel produced. The market is being flooded with crude glycerol; as a result its economic value has gone down. It is clear that new uses for biodiesel by-product crude glycerol are needed and the literature shows a promising result to use as a feedstock for steam reforming to produce hydrogen.

Feedstock for reforming

Biodiesel by-product, a low quality crude glycerol can be the feedstock for steam reforming to produce hydrogen. This by-product may vary depending upon the starting material for the biodiesel production. Biodiesel is generally produced from virgin oil, waste vegetable oil, animal fats, algae and other biomass.

Crude glycerol composition

The crude glycerol produced from biodiesel production is of low quality and has low economic value. The impurities such as methanol, ash, water, and fatty matter makes it of low quality (Slinn, 2008). Biodiesel production needs excess methanol during transesterification which will form a layer on glycerol. Likewise, free fatty acids present in the initial feedstock can react with the base to form soaps (fatty matter) and are soluble in glycerol⁵. The feedstock, by-product glycerol, for my research composes of 40.4% fatty matter, 33.1% Glycerol, 23.3% methanol, 3.2% water (Slinn, 2008).

Steam reforming process

Steam reforming is a method of producing hydrogen from hydrocarbons. It is most common and least expensive method of producing commercial bulk hydrogen. In this method steam reacts with hydrocarbons at high temperature (700 -1100°C) and in the presence of a catalyst to yield hydrogen and carbon monoxide. Carbon monoxide is then subjected to the water-gas shift (WGS) reaction to produce more hydrogen. The various factors that affect the yield of hydrogen are basically temperature and feed steam/carbon (S/C) ratio (see figure 3 and 4) (Slinn, 2008).

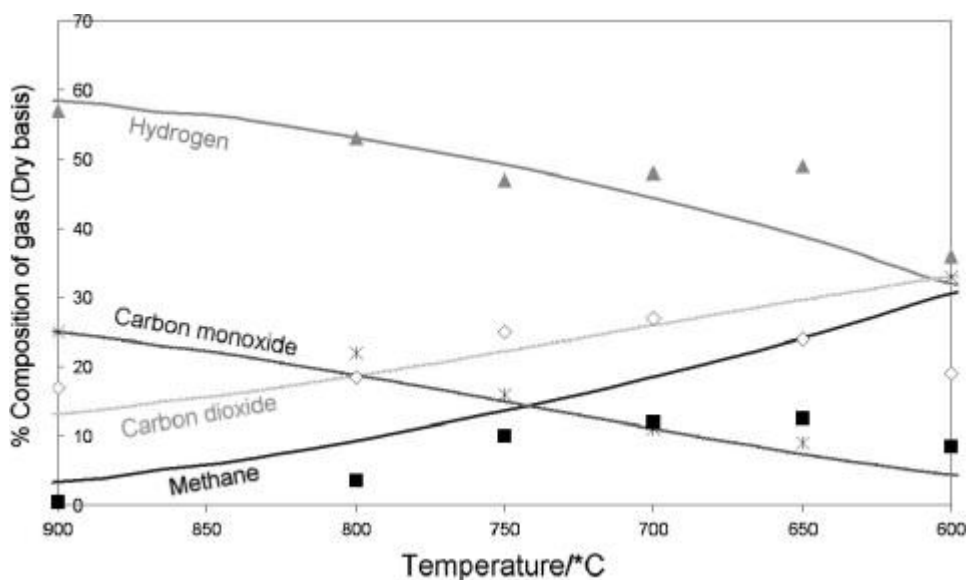


Fig.3. Effect of temperature on product gas stream composition (Slinn, 2008).

Key to Symbols: Hydrogen, (▲); Carbon dioxide, (◆); Methane, (■); Carbon monoxide, (⋈) $S/C = 1.35$

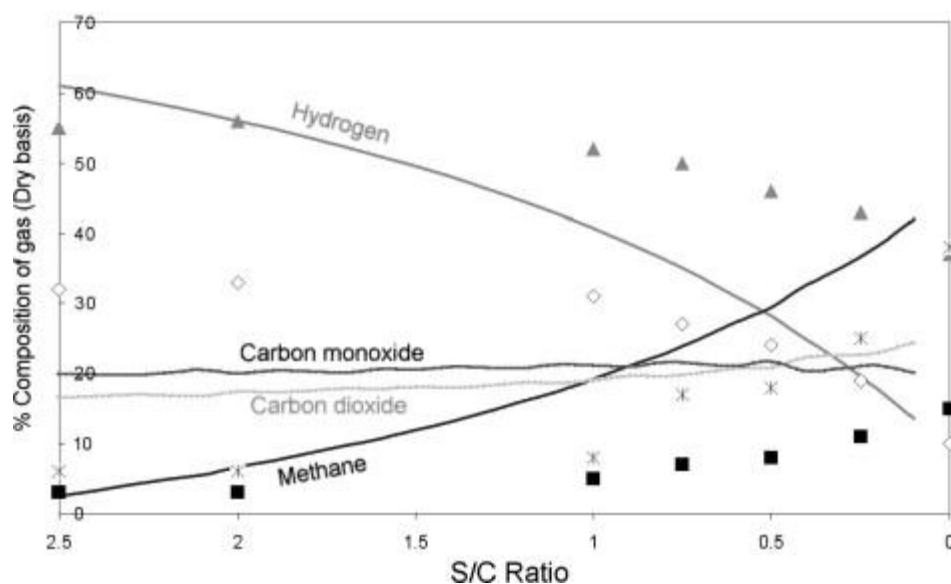
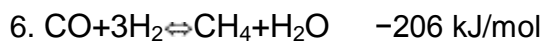
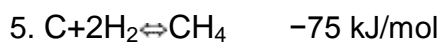
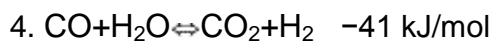
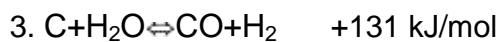
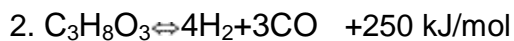
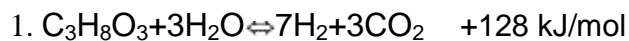


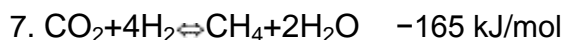
Fig.4. Effects of feed S/C ratio on product gas stream composition (Slinn, 2008).

Key to Symbols: Hydrogen, (▲); carbon dioxide, (◆); methane, (■); carbon monoxide, (⋈) Temperature = 850 °C

Chemical reactions

There are two main reactions which can occur in a high temperature steam and hydrocarbon mixture; steam reforming and pyrolysis. However many side reactions occur simultaneously on the reformer including many side reactions as shown below.





Adapted from Slinn (2008)

Uses of hydrogen

Hydrogen has a wide application in industry for refining, treating metals, and processing fuels. It is also used to make fertilizers, glass, vitamins, cosmetics, soaps, lubricants, cleaners, and semiconductor circuits. It can be used to make electricity. NASA is the primary user of hydrogen as energy fuel-called fuel cells- to power the shuttle's electrical system (Hydrogen Energy, 2008). Hydrogen can fuel tomorrow's fuel-cell vehicles if we can produce large amount of hydrogen. Hydrogen can also replace today's natural gas for heating and cooling homes and powering hot water heaters⁶. Hence, if hydrogen is produced from waste glycerol, it's a win-win situation because biodiesel industry can boom which makes less dependable on foreign oil and by-product waste glycerol can turn into valuable product and solve energy crisis.

Problem statement

When biodiesel by-product glycerol is heated in presence of catalyst and steam at 850°C and 1 atm, glycerol instantly converts to hydrogen and carbon monoxide. This method is called steam reforming of glycerol. Carbon monoxide produced as a by-product of steam reforming is further subjected to water gas shift (WGS) reaction where it reacts with excess steam in presence of catalyst to form hydrogen and carbon dioxide. In general,

Co/MgO, Co/Al₂O₃, Ni/MgO is used as catalyst for steam reforming because they are cheaper and easily available.

During the reaction, other biodiesel by-product impurities mainly methanol and fatty matter would also steam reform. Methanol reforms to give the same yield as glycerol does whereas fatty matter are harder to reform and are likely to form more carbon deposition on catalyst and on the reactor surface. Hydrogen produced from this process contains impurities such as carbon monoxide, carbon dioxide and methane. Hence, depending on use, it requires further purification.

CHAPTER II

METHODS

The overall goal of this research is to design a steam reforming process to produce hydrogen from biodiesel by-product glycerol. The following are the methods for the process design:

- Literature search on pure and waste biodiesel by-product glycerol, steam reforming, water-gas shift (WGS) reaction, and purification process
- Flowsheet design as shown in figure 5
- Calculation for conversion of glycerol into hydrogen and other by-product gases based on the literature
- Process simulation on ASPEN Plus and selection of appropriate thermodynamic data
- Identifying opportunities for process integration and cost minimization
- Cost evaluation of the process and sensitivity analysis

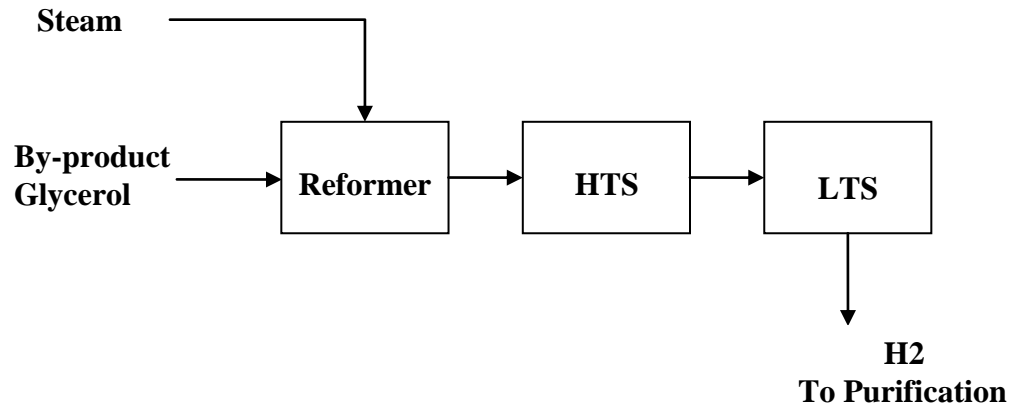


Fig.5. Design process to produce hydrogen.

Glycerol conversion

During the experiment with by-product glycerol, the longest run under constant operating conditions was reached in 10 hours for complete conversion (Slinn, 2008). In order to make it feasible for steam reforming process in large scale, the amount of glycerol converted was calculated to be 68% in the interval of 4 minutes period as shown reaction kinetics.

Kinetics

From the literature review:

- 99% experimental conversion of glycerol Hence, $C_{out} = .01 C_{in}$
- Residence time interval for the conversion, $(t) = 10$ hrs

The reaction rate can be calculated with the relation shown in eq.1.

Equation 1. Relation between concentration and the reaction rate

$$\frac{C_{out}}{C_{in}} = e^{-kt} \quad (1)$$

Where, k = Reaction Rate

C = Concentration

T = Time interval

Therefore, reaction rate is calculated as 0.007675.

The following properties of by-product glycerol and assumption are made for the process simulation in the context of the biodiesel production industries

- The feedstock consists of 33% of glycerol. Hence, inlet concentration (C_{in}) is 0.33.
- Assuming residence time = 4 minute.

Using Eq. 1 and calculated reaction rate, outlet concentration (C_{out}) is calculated as 0.32.

Hence, glycerol conversion is 68%.

Process design

Process simulation is done on ASPEN PLUS Simulator to determine how different compound interact with each other and how the reaction yield is separated to get pure hydrogen. The simulation consists of various different processes such as steam reforming, water gas shift reaction, and purification of hydrogen as shown in design flowsheet.

Feedstock specifications

A biodiesel process plant capacity of producing 40 million gallons of biodiesel per year produces about 40 pound moles per hour of crude glycerol (He, 2005). The feedstock

consists of 40.4% fatty matter, 33.1% Glycerol, 23.3% methanol, 3.2% water (Slinn, 2008). The inlet temperature and pressure is considered as 60oC and 1 atm respectively.

Table.1 shows the composition and the flow rate of the inlet feed.

Table 1. Feed stock composition and molar flow rate

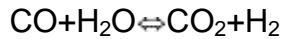
Inlet Compositions	Compositions %	Flow rate (lbmol/hr)
Methanol	23.3	9.2
Water	3.2	1.6
Glycerol	23.3	13.2
Fatty matter (Oleic acid)	40.4	16
Total	100	40
Steam	100	50

Steam reforming process

Steam reforming process consists of a stoichometer reactor (REFORMER) where the feed stock is heated at 850°C and 1atm in presence of superheated steam. Under this constant operating condition for 4 minutes of batch process, 68% of crude glycerol and 100% methanol converted into gaseous mixture where as fatty matter burns forming a carbon deposition on the reactor surface. This carbon deposition helps to maintain the reactor temperature constant at 850°C and requires cleaning after certain period of time depending upon the layer of the carbon formed. Unreacted 32% glycerol is recycled back into the reactor as a feedstock for steam reforming.

Water gas shift (WGS) reaction

The water-gas shift (WGS) reaction is to convert carbon monoxide produced from steam reforming into hydrogen in presence of steam and catalyst as shown in the reaction below.



Adapted from (Slinn, 2008)

The catalytic water-gas shift reaction is initially carried out in a high temperature shift (HTS) reactor at 350°C. To achieve higher conversions of CO to H₂, the gas leaving HTS reactor is cooled to 210°C and passed through a low temperature shift (LTS) reactor. Approximately 90% of the CO is converted to H₂ in the first HTS reactor and 90% of the remaining CO is converted in the LTS reactor.

Thermodynamic

RK-Soave thermodynamic properties were used in the simulation. Not all the thermodynamic data needed were incorporated in ASPEN PLUS and hence some data were entered by user-defined method. The fatty matter was entered as oleic acid and its molecular structure was imported to the ASPEN.

CHAPTER III

RESULTS

Steam reforming process followed by water gas reaction seems to be a good method for converting biodiesel by-product waste glycerol into valuable product. The process is sole function of reaction temperature. Higher the temperature, more glycerol reforms producing more hydrogen. At 850°C maintained in a reactor would convert most of the glycerol into gaseous product. During simulation standard atmospheric pressure was chosen to make this process economic friendly. Figure 6 shows the process design flow sheet on Aspen PLUS simulator. The results from the simulator are attached as 3.3 Aspen PLUS Result Summery.

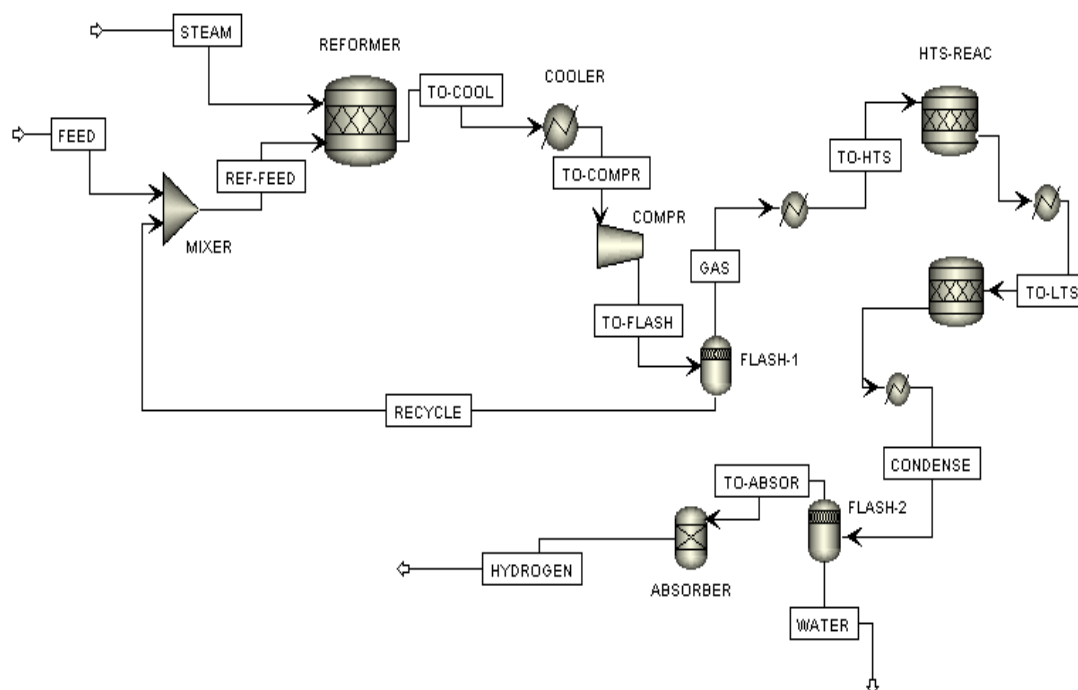


Fig.6. Process flow sheet.

Results from steam reforming process

The product stream from steam reforming process consists of gaseous mixture, unreacted excess glycerol, and by-product such as carbon tar and water. Gaseous mixture is further subjected to WGS reaction and then purification process. Carbon tar is produced from the Oleic acid which is hard to reform under the conditions of 850°C and 1 atm. At 850°C carbon monoxide can react with hydrogen to produce methane. Hence, additional separation process is required for separating methane. Table 2 summarizes the result from the reforming process.

Table 2. Results from Steam Reforming Process

Product Component	Mole Flow (lbmol/hr)	Composition %
Excess Glycerol	11	3
Water	20	5
Oleic Acid	40	11
Hydrogen	219	58
Methane	0	0
Carbon Monoxide	7	2
Carbon Dioxide	84	22
Total	381	100

Results from water gas shift reaction

In this process, 7 lbmol/hr carbon monoxide produced from steam reforming is converted to produce hydrogen. As a result, carbon dioxide gas is produced as a reaction by-product. After WGS reaction the gaseous stream contains some trace of carbon monoxide which is subjected to the further purification process. Table 3 summarizes the result from the WGS reaction process.

Table 3. Results from Water Gas Shift Reaction

Product Component	Mole Flow (lbmol/hr)	Composition %
Excess Glycerol	2	0
Water	14	4
Oleic Acid	0	0
Hydrogen	225	68
Methane	0	0
Carbon Monoxide	1	0
Carbon Dioxide	90	27
Total	330	100

Aspen PLUS result summary

	S1	S2	S3	S4	S5	S6	S7
Temperature F	140.0	1589.0	254.4	254.4	1562.0	1562.0	1562.0
Pressure psi	14.70	14.70	14.70	14.70	14.70	14.70	14.70
Vapor Frac	0.000	1.000	0.594	0.594	1.000	1.000	1.000
Mole Flow lbmol/hr	100.000	100.000	180.000	20.000	301.176	33.464	380.640
Mass Flow lb/hr	15146.827	1801.528	15253.520	1694.835	15253.520	1694.835	16948.355
Volume Flow cuft/hr	273.849	149590.702	55596.116	6177.346	444901.979	49429.897	562277.933
Enthalpy MMBtu/hr	-25.769	-9.029	-31.317	-3.480	-15.050	-1.575	-13.916
Mole Flow lbmol/hr							
METHANOL	23.000		20.700	2.300	20.700	2.300	
WATER	4.000	100.000	93.600	10.400	33.012	10.400	20.412
GLYCEROL	33.000		29.700	3.300	9.504	1.056	10.560
OLEIC-01	40.000		36.000	4.000	36.000	4.000	40.000
HYDROGEN					141.372	8.976	219.348
METHANE							
CARBO-01						6.732	6.732
CARBO-02					60.588		83.588
	S8	S9	S10	S11	S12	S13	S14
Temperature F	1562.0	1562.0	680.0	1974.7	400.0	400.0	680.0
Pressure psi	14.70	14.70	14.70	308.61	308.61	308.61	308.61
Vapor Frac	1.000	1.000	1.000	1.000	1.000	0.000	1.000
Mole Flow lbmol/hr	340.640	40.000	340.640	340.640	330.895	9.745	330.895
Mass Flow lb/hr	5649.685	11298.670	5649.685	5649.685	4803.321	846.364	4803.321
Volume Flow cuft/hr	502999.499	59012.275	283580.163	28951.080	9960.701	12.132	13209.116
Enthalpy MMBtu/hr	-14.399	-1.922	-17.396	-12.874	-16.048	-2.494	-15.277
Mole Flow lbmol/hr							
METHANOL							
WATER	20.412		20.412	20.412	19.764	0.648	19.764
GLYCEROL	10.560		10.560	10.560	1.520	9.040	1.520
OLEIC-01		40.000					
HYDROGEN	219.348		219.348	219.348	219.340	0.008	219.340
METHANE							
CARBO-01	6.732		6.732	6.732	6.732	< 0.001	6.732
CARBO-02	83.588		83.588	83.588	83.540	0.048	83.540
	S15	S16	S17	S18	S19	S20	S21
Temperature F	680.0	410.0	410.0	77.0	32.0	32.0	32.0
Pressure psi	308.61	308.61	308.61	308.00	308.00	308.00	308.00
Vapor Frac	1.000	1.000	1.000	0.957	1.000	0.000	1.000
Mole Flow lbmol/hr	330.895	330.895	330.895	330.895	316.332	14.563	90.328
Mass Flow lb/hr	4803.321	4803.321	4803.321	4803.321	4428.071	375.249	3972.474
Volume Flow cuft/hr	13210.200	10001.165	10001.530	5930.461	5410.774	5.352	1304.811
Enthalpy MMBtu/hr	-15.377	-16.124	-16.135	-17.316	-15.390	-2.056	-15.348
Mole Flow lbmol/hr							
METHANOL							
WATER	13.706	13.706	13.100	13.100	0.068	13.032	0.068
GLYCEROL	1.520	1.520	1.520	1.520	trace	1.520	trace
OLEIC-01							
HYDROGEN	225.398	225.398	226.004	226.004	226.004	< 0.001	
METHANE							
CARBO-01	0.673	0.673	0.067	0.067	0.067	trace	0.067
CARBO-02	89.599	89.599	90.204	90.204	90.193	0.012	90.193

S22

Temperature F	32.0
Pressure psi	308.00
Vapor Frac	1.000
Mole Flow lbmol/hr	226.004
Mass Flow lb/hr	455.597
Volume Flow cuft/hr	3924.114
Enthalpy MMBtu/hr	-0.068
Mole Flow lbmol/hr	
METHANOL	
WATER	
GLYCEROL	
OLEIC-01	
HYDROGEN	226.004
METHANE	
CARBO-01	
CARBO-02	

Separation process

During steam reforming process, there are unreacted glycerol, water, and carbon tar. Gaseous product is in the mixture form and requires purification to produce pure hydrogen. Hence, it involves various separation processes such as flashing and absorption. Carbon tar is formed on the surface of the reactor as a fouling and cleaning is required after certain time interval.

Removal of glycerol

Excess glycerol is separated by flashing which is recycled back into the process. In this process, the compressed exit stream (S11, TO-FLASH) from compressor is sent to the flash tank (FLASH-1). This flash tank operates at 205°C and 21 atm along with RK-SOAVE thermodynamic properties. About 86% of excess glycerol is separated from gaseous stream and send to the reactor as a feedstock.

Removal of water

Water is removed by condensation process. In this process, the stream coming out of the WGS reaction is cooled down to the room temperature (25°C) and sent to the condenser. About 99% of water and the remaining glycerol from FLASH2-1 are separated. The gaseous stream is passed into the absorption unit to separate CO₂. The final product, hydrogen, produced through this process contains traces of carbon oxides and methane and requires further purification depending upon its use. Figure 7 shows the schematic of the process, recycle, and separation.

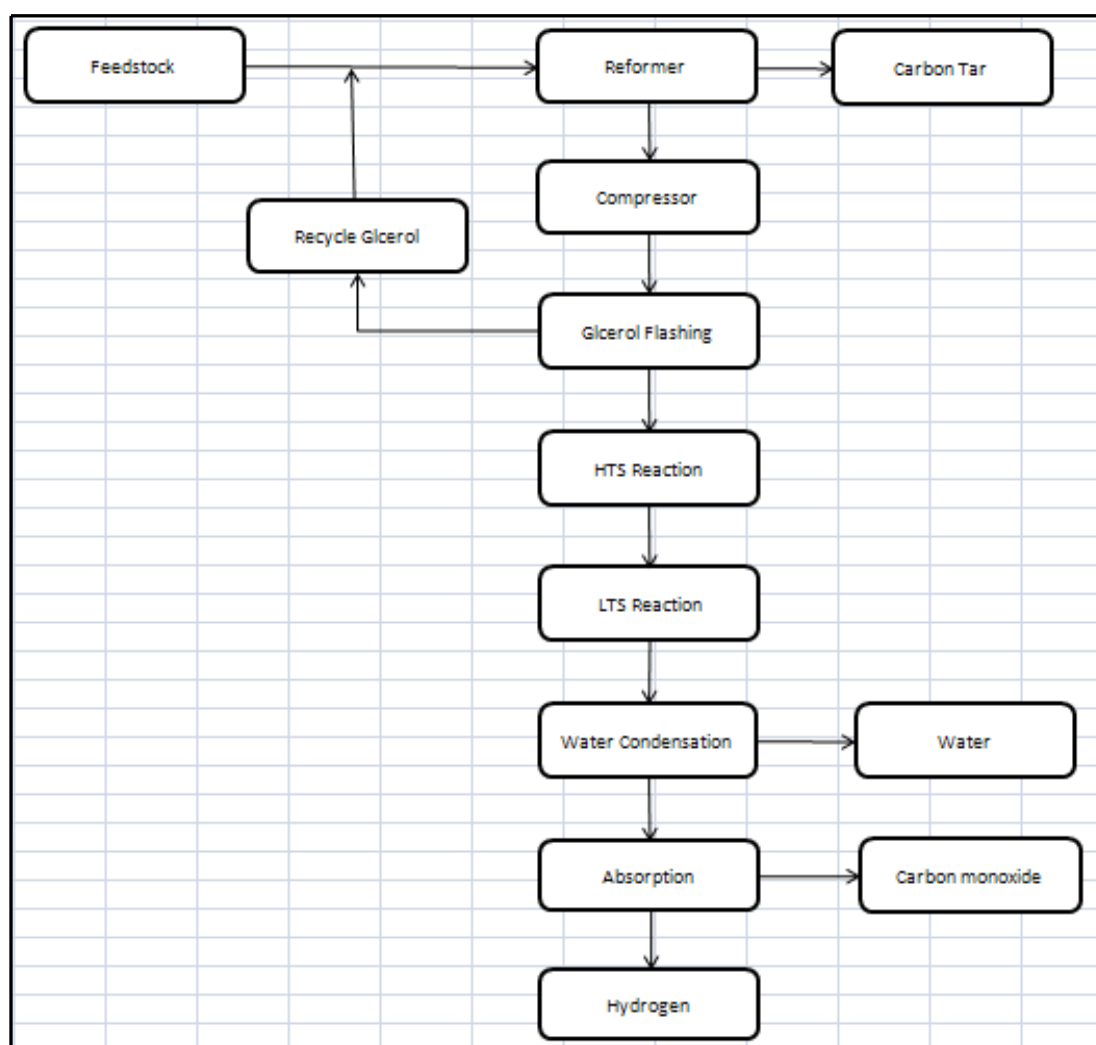


Fig.7 Schematic flow diagram for separation.

CHAPTER IV

CONCLUSIONS

Steam reforming is a viable alternative use for by-product waste glycerol. The design consists of steam reforming followed by WGS reaction and then hydrogen purification. This process could potentially take all the waste from the biodiesel plant and convert to sellable energy. If a waste can be converted to renewable hydrogen, it can boom the biodiesel industry and can make less dependable on foreign oil.

Hydrogen has a wide application in industries and refineries. In the United States, about 17.2 billion pounds of hydrogen are produced per year and 95% are from steam reforming of methane (Hydrogen Now). It can be used as a fuel in tomorrow's fuel-cell vehicles if produced in a large amount. The major challenge of the process could be green house gaseous emission. The mass of CO₂ emitted is more than twice than the mass of hydrogen produced. Hence, the future research work includes proper disposal methods for CO₂ and cost analysis of the design.

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