

DEVELOPMENT OF CEMENT KILN DUST (CKD)
BASED CATALYST FOR BIODIESEL
PRODUCTION FROM WASTE COOKING OIL

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DEVELOPMENT OF CEMENT KILN DUST (CKD) BASED CATALYST FOR
BIODIESEL PRODUCTION FROM WASTE COOKING OIL

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Thesis submitted in fulfillment of the requirements for the award of the degree of
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SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of Bachelor of Chemical Engineering.

Signature

Name of Supervisor: DR.JOLIUS BIN GIMBUN

Position:

Date: 20 January 2012

STUDENTS'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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Date: 20 January 2012

*Specially dedicated to my beloved family, my brothers and sisters from “GEMURUH”
circle team, and all my friends.*

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ABSTRACT

Cement kiln dust (CKD) is alkaline materials which produce as a by-side product in the cement manufacturing process which contain amount of alkalis like Na_2O , K_2O and large amount of free lime. The price of catalyst derived from noble metals such as platinum is expensive and this will drive the price of biodiesel even higher than the dinodiesel. Therefore, it is necessary to synthesize a cheaper catalyst to make the production of biodiesel competitive and feasible. In this work, CKD will be utilised as raw material to produce catalyst for transesterification of waste cooking oil. This CKD based catalyst was prepared by impregnated with the potassium hydroxide and the other by methanol and water. The catalyst was calcined at $650\text{ }^{\circ}\text{C}$ for 3 hours. The transesterification process of waste cooking oil with methanol was used to investigate the yield of biodiesel. The catalysts were characterized using pH measurement, Thermogravimetric analysis (TGA) and Fourier transform infrared (FTIR). The experimental result showed that a CKD/KOH give the highest purity and yield of biodiesel compared to the other catalyst which is 97.85% and 89.34%. The higher alkalinity gives the higher purity and yield. This CK/KOH catalyst is the best catalyst compared to CKD, CKD/ CH_3OH and CKD/ H_2O for the biodiesel production. The catalyst can be produce with a low cost of material and high efficiency of production.

ABSTRAK

Habuk tanur simen (CKD) adalah bahan-bahan alkali yang menghasilkan sebagai produk sampingan dalam proses pembuatan simen yang mengandung jumlah alkali seperti Na_2O , K_2O dan jumlah kapur yang besar. Harga pemangkin yang berasal dari logam seperti platinum adalah mahal dan ini akan mendorong harga biodiesel lebih tinggi daripada dinodiesel. Oleh itu, keperluan untuk mensintesis satu pemangkin yang lebih murah untuk membuat pengeluaran biodiesel berdaya saing dan boleh dilaksanakan. Dalam kerja lapangan ini, CKD akan digunakan sebagai bahan mentah untuk menghasilkan pemangkin untuk transesterification sisa minyak masak. Ini pemangkin berasaskan CKD telah disediakan oleh impregnated dengan hidroksida kalium, methanol dan air. Pemangkin calcined pada suhu $650\text{ }^\circ\text{C}$ selama 3 jam. Proses transesterification sisa minyak masak dengan metanol telah digunakan untuk menyiasat hasil biodiesel. Pemangkin dicirikan menggunakan pengukuran pH, Thermogravimetric analisis (TGA) dan Fourier transform infrared (FTIR). Hasil uji kaji menunjukkan bahawa CKD / KOH memberikan ketulenan tertinggi dan hasil biodiesel berbanding pemangkin lain iaitu 97,85% dan 89,34%. Kealkalian yang tinggi memberikan ketulenan dan hasil yang lebih tinggi. Ini pemangkin CK/KOH adalah pemangkin terbaik berbanding dengan CKD, CKD/ CH_3OH dan CKD/ H_2O untuk pengeluaran biodiesel. Pemangkin ini boleh menghasilkan dengan kos bahan mentah yang rendah dan kecekapan pengeluaran yang tinggi.

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LIST OF SYMBOLS

%	Percentage
US \$	United States Dollar
<	Less than
°C	Degree Celsius
μ	Micro

LIST OF ABBREVIATIONS

CKD	Cement Kiln Dust
cm	centimeter
cm ⁻¹	per centimeter
FAEE	Fatty acid ethyl esters
FAME	Fatty Acid methyl esters
FT-IR	Fourier Transform Infrared Spectrometry
GC MS	Gas chromatography with a mass selective detector
h	hour
L	litre
mg	miligram
ml	mililiter
mm	milimeter
rpm	revolution per minutes
TGA	Thermogravimetric
U.S.	United States
USEPA	United States Environmental Pollution Agency
WCO	Waste cooking oil

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Cement kiln dust (CKD) is alkaline materials which produce as a by-side product in the cement manufacturing process. CKD is a fine-grained solid and is a highly alkaline waste material that is removed from cement kiln exhaust gas. This material is consisting of fine particles gathered by dust collection system during the cement manufacturing process. (Mackie et al., 2009; Konsta-Gdoutos and Shah, 2003; Peethamparan et al., 2008).

In general, there are some elements in the CKD such as Ca, Fe and Sr. (Polat et al., 2004). All CKD mainly contains amount of alkalis (Na_2O , K_2O) and sulfate besides contain a large amount of free lime. Free lime in the CKD can make it a substitute for fertilizers and stabilizing wastewater streams (Konsta-Gdoutos and Shah, 2003).

In the recent survey in the United State showed, there are 87 million tonnes of cement clinker produced in 2006, 1.2 million tonnes of CKD were reused on or off-site (not including recycling into kiln feed) while 1.4 million tonnes were landfilled. In addition, 0.3 million tonnes of CKD was reclaimed from landfills, mainly for use as kiln feed (Mackie et al., 2009). The higher alkalinity and finer particle size in their properties make CKD usable for several applications such as waste solidification, replacement of

Portland cement in concrete block manufacturing, construction of hydraulic barriers (Peethamparan et al., 2008) also as a catalyst (Lin et al., 2011).

CKD has been found to be efficient, inexpensive and environmental friendly catalyst for biodiesel production (Lin et al., 2011). The combination of this CKD and WCO for biodiesel production makes it more inexpensive and worth. It is reported that approximately 70%-85% of the total biodiesel production cost arises from the cost of the raw material (Fatimah, 2009). WCO is easy to be found everywhere in the world, which have a large amount of waste lipids generated from restaurants, food processing industries and fast food shops everyday (Fatimah, 2009).

1.2 PROBLEM STATEMENT

There are a big number of wastes CKD in the United States that was researched by Mackie et al. (2010) which is about 87 millions of CKD was generated in 2006. Most of this waste reused as a soil or clay stabilization, agricultural soil amendment, concrete products and etc (Adaska et al., 2008).

CKD is also suitable used as a catalyst for biodiesel production which can improve the performance of biodiesel production (Lin et al., 2011). CKDs are widely available and typically available at no cost compared to other catalyst like platinum. It can be alternative as a catalyst. The component of CKD contains of silica, calcium carbonate, and calcium oxide “free lime” (Peethamparan et al., 2008) which is suitable as a catalyst to produce the biodiesel in the high performance of production.

Currently, the high cost of biodiesel is the major blockage for biodiesel to commercialize. Biodiesel usually costs over US\$0.5/l, compared to US\$0.35/l for petroleum based diesel. It is reported that the high cost of biodiesel is mainly due to the cost of virgin vegetable oil (Zhang et al., 2003).

The use of waste cooking oil replace the virgin oil to produce biodiesel is an effective way to reduce the raw material cost because it is estimated about half of the price of virgin oil. In addition, using waste cooking oil could also help to solve the problem of waste oil disposal (Zhang et al., 2003).

1.3 OBJECTIVE OF STUDY

The objective of this research is to develop a CKD based catalyst for biodiesel production from waste cooking oil (WCO) transesterification using various activation methods.

1.4 SCOPE OF RESEARCH

In order to achieve the objective of this research, which is to develop a CKD based catalyst for waste cooking oil (WCO) transesterification using various impregnation alkali solutions, the scope of study was divided into two main parts as following:

1. To characterize the CKD in term of pH of the catalyst with the pH meter, functional group of elements with Fourier Transform Infrared Spectrometry (FTIR) and the change in weight with the relation change in temperature using Thermogravimetric analysis (TGA).
2. To evaluate the catalyst performance using transesterification process of biodiesel from waste cooking oil.

1.5 RATIONAL AND SIGNIFICANT

The purpose of this study is to produce CKD waste as catalyst for biodiesel production from WCO. As knowing in the Adaska et al. (2008) mentioned that the CKD production was used as clay stabilization, agricultural soil amendment, concrete products and etc. The catalyst that have been used for biodiesel production before like glucose–

starch mixture (Chen et al., 2011) and metal oxide (Zabeti et al., 2009). The significant to produce CKD as a catalyst because it has low material cost compare to the other catalyst like platinum and etc because it get from the waste of cement production.

CHAPTER 2

LITERATURE REVIEW

2.1 CEMENT KILN DUST

2.1.1 Overview of cement kiln dust

Cement manufacturing is an important manufacturing throughout the world. United States plant produces 99.8 million metric tons of cement in 2006. Cement kiln dust (CKD) is a by-product material of the cement manufacturing process. The byproduct industrial and waste materials must be managed responsibly to ensure a clean and safe environment. Over the past several years, the management and uses of cement kiln dust was increasing dramatically, thus reducing its dependency on landfill disposal (Adaska et al., 2008).

Cement kiln dust is produced in the kiln during the production of cement clinker. The dust is a particulate mixture of some calcined and unreacted raw feed. All particulates are captured by the exhaust gases and then collected in particular matter control devices such as cyclones, bag houses and electrostatic precipitators (Adaska et al., 2008). Cement manufacturing process is shown in the **Figure 2.1**.

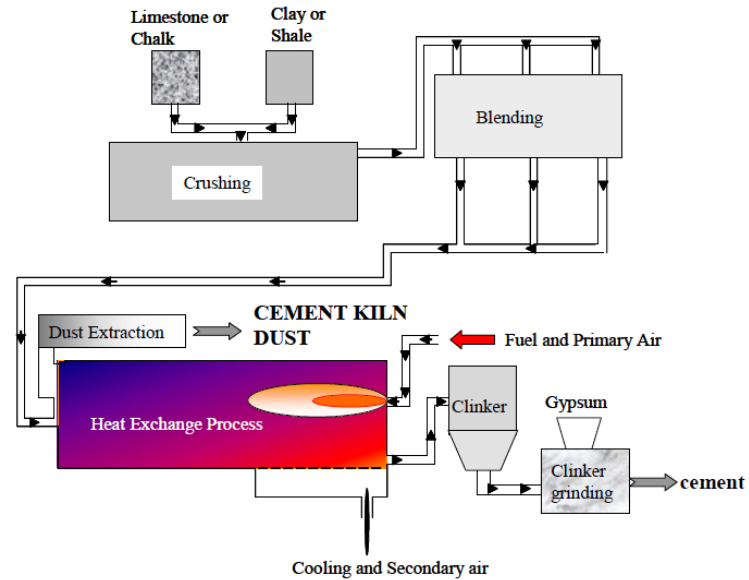


Figure 2.1: Cement manufacturing process (Sreekrishnavilasam and Santagata, 2006)

The chemical and composition content of CKD depends on the raw materials, plant configuration, and the processing type of cement production (Sreekrishnavilasam and Santagata, 2006). **Table 2.1** shows some typical composition of cement kiln dust in general.

Table 2.1: Typical composition of cement kiln dust by Haynes and Kramer (1982)

Constituent	% by weight	Constituent	% by weight
CaCO ₃	55.5	Fe ₂ O ₃	2.1
SiO ₂	13.6	KCl	1.4
CaO	8.1	MgO	1.3
K ₂ SO ₄	5.9	Na ₂ SO ₄	1.3
CaSO ₄	5.2	KF	0.4
Al ₂ O ₃	4.5	Others	0.7

2.1.2 Cement Kiln dust disposal

Cement industry has established the Cement Manufacturing Sustainability (CMS) Program to balance society's need for cement products. The major element in the CMS programmed is to establish the Environmental Performance Measures. In the case of CKD in the U.S. cement industry, at year 2020, 60 percent reduction (from a 1990 baseline) in the amount of cement kiln dust disposed per ton of clinker produced (Sreekrishnavilasa and Santagata, 2006). **Figure 2.2** shows the flow chart for gross CKD management practices in the United States and **Table 2.2** shows the states with the Highest Amount of CKD Used for Beneficial applications and is currently developing a new CKD reduction goal (Adaska et al., 2008).

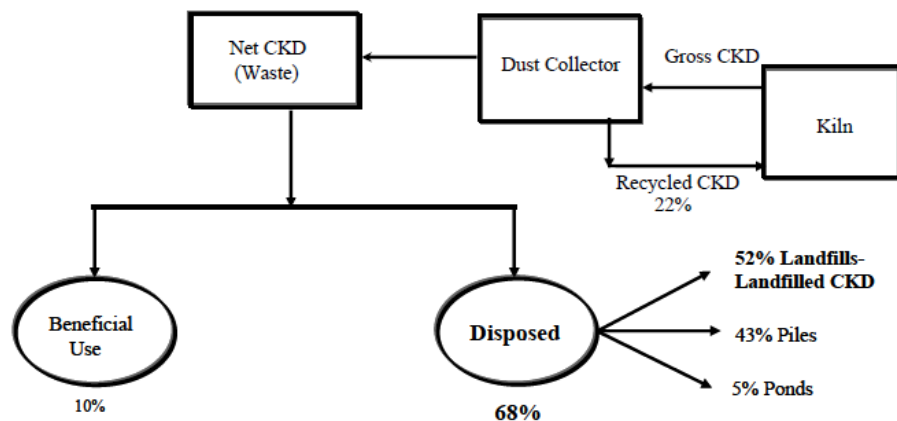


Figure 2.2: Flow chart for gross CKD management practices in the United States (Sreekrishnavilasam and Santagata, 2006)

In the United States more than four million tons of CKD that are unsuitable for recycling in the cement manufacturing process, require disposal annually. United States Environmental Pollution Agency (USEPA, 1993) estimated that, 52% was disposed in landfills, 43% percent in piles, and less than 5% in ponds. The average piles were 15 m thick. Maximum reported thickness for CKD landfills and waste piles were 56.4 m and 34.6m, respectively.

Table 2.2: The states with the Highest Amount of CKD Used for Beneficial applications

State	Quantity of CKD beneficially reused, metric tons	State	Quantity of CKD beneficially reused, metric tons
Oklahoma	154,477	Indiana	82,325
Texas	144,043	California	66,801
Pennsylvania	102,760	Arkansas	61,990
Ohio	86,453	Maryland	50,562
Illinois	85,330	Missouri	48,250

The amount of CKD used for beneficial applications has been increased dramatically over the 16 years. Annual use of CKD for beneficial applications has ranged from a low of 574,800 metric tons to 1.16 million metric tons. **Table 2.3** shows the historical cement kiln dust production and management (Adaska et al., 2008).

Table 2.3: Historical cement kiln dust production and management

Year	Plants responding to survey for given year	CKD beneficially reused on or off site, metric tons	CKD sent to landfill, metric tons	CKD reclaimed from landfilled, metric tons	Annual clinker production, metric tons	CKD sent to a landfill/clinker produced, kilograms / metric tons
1990	84	752,152	2,655,725	No data	44,360,364	60
1995	94	651,205	3,146,952	No data	61,729,315	51
1998	95	768,601	2,499,651	13,409	67,104,547	37
2000	92	574,803	2,223,190	79,171	68,263,086	33
2001	102	924,552	2,329,132	231,904	75,683,170	31
2002	101	664,848	1,989,680	103,223	77,636,598	26
2003	102	718,410	1,995,143	116,416	79,356,511	25
2004	102	917,968	1,993,421	69,099	83,945,430	24
2005	102	987,717	1,429,150	104,952	85,568,243	17
2006	101	1,160,011	1,403,062	261,351	86,686,834	16

2.2 CATALYST

A catalyst is a substance which changes the rate of a chemical reaction but is chemically unchanged at the end of the reaction. While most catalysts make the rate of chemical reactions go faster, but some can slow down the chemical rate. A catalyst can also make a chemical reaction possible that would not otherwise be.

Catalyst used in the transesterification of triglycerides can be classified as homogeneous and heterogeneous catalyst. Fatimah (2008) stated that excess amount of catalyst would lead to the higher amount of production cost and reduce the product yield.

2.1.1 Homogeneous Catalyst

Homogeneous catalyst is the process which involves at least one of the reactant. Basically, in this transesterification process, there are two types of homogeneous catalyst which is acid catalyst and alkali catalyst. Homogeneous basic catalyst provides much faster reaction rates than heterogeneous catalyst, but it is difficult to separate homogeneous catalyst from the reaction mixture (Fatimah, 2008).

2.1.2 Heterogeneous Catalyst

A heterogeneous catalytic is the process that involves more than one phase, usually the catalyst is a solid and the reactant and product are in liquid or gaseous form. There are many advantages of using heterogeneous catalyst such as non-corrosive, environmental friendly, fewer disposal problems, easier in separation from liquid product and they can be design to give higher activity, selectivity and longer catalyst lifetime. Example of heterogeneous catalyst such as alkaline earth metal oxides, anion exchange resins and various alkali metal compounds supported on alumina and that can be use in various type of chemical reaction including transesterification (Fatimah, 2008).

2.3 BIODIESEL

2.3.1 Background of Biodiesel

Biodiesel is known as an alternative diesel fuel especially for substitute diesel in developed countries mainly for transportation and agriculture industries. In recent years, biodiesel are more important due to insufficient of petroleum fuel and the needs of environmental friendly energy resources. Biodiesel is a renewable energy sources that are made from natural vegetable oil, animal fats, or singles cell oil (Ghadafi, 2008). However, the cost of biodiesel is high cause to the high cost of raw material (about 70-75% of the total cost) (Jasrina, 2008).

There are many ways how biodiesel roles of environment benefits more than fossil diesel. One of the key aspects of the life cycle assessment is a global warming potential, expressed as carbon dioxide, equivalent to CO₂.CO₂ is produced during the whole production process of fuels. According to the positive energy balance of biodiesel and the fact that biodiesel consists of renewable material one could expect a large saving of greenhouse gases compared to fossil fuel (Angela, 2009).

Nowadays, with the price of crude fossil fuel prices is too high, biodiesel have emerged as the fastest growing industries worldwide. Several countries especially United State and European Union are fully supporting the production of biodiesel from the agriculture sector. In year 2006, approximately 6.5 billion liters of biodiesel was produced globally. However, expected by the year 2020, biodiesel production from Brazil, China, India and some South East Asia countries such as Malaysia and Indonesia could contribute as much as 20% of production (Lam et al., 2010).

2.3.2 Composition of Biodiesel

Biodiesel is a mixture of fatty acid alkyl esters. There will be a mixture of fatty acid methyl esters (FAME) when methanol is used as reactant and if ethanol is used as reactant, the mixture will be fatty acid ethyl esters (FAEE). However, methanol is commonly used in biodiesel production because of their low cost and availability. Based on different feedstock, the biodiesel produced will have the different composition of FAME like in **Table 2.4** have shown below (Lam et al., 2010).

Table 2.4: Typical fatty acid composition (%) for different common oil source

Fatty acid	Soybean	Cottonseed	Palm	Lard	Tallow	Coconut
Lauric (C12:0)	0.1	0.1	0.1	0.1	0.1	46.5
Myristic (C14:0)	0.1	0.7	1.0	1.4	0.8	19.2
Palmitic (C16:0)	0.2	20.1	42.8	23.6	23.3	9.8
Stearic (C18:0)	3.7	2.6	4.5	14.2	19.4	3.0
Oleic (C18:1)	22.8	19.2	40.5	44.2	42.4	6.9
Linoleic (C18:2)	53.7	55.2	10.1	10.7	10.7	2.2
Linolenic (C18:3)	8.6	0.6	0.2	0.4	0.4	0.0

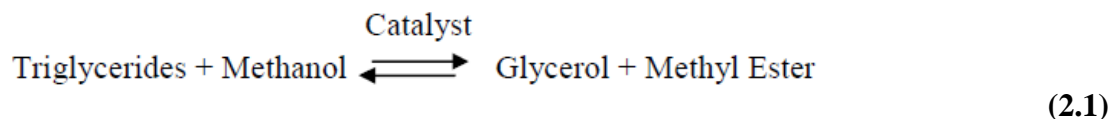
According to Lam et al. (2010) there are some of chemical structures of fatty acid methyl ester that will be found in the transesterification reaction of biodiesel production as shown in **Figure 2.5**.

Table 2.5: Chemical structure of common FAME

Methyl ester	Formula	Common acronym	Molecular weight
Methy palmitic	$C_{17}H_{34}O_2$	C16:0	270.46
Methy stearate	$C_{19}H_{38}O_2$	C18:0	298.51
Methy oleate	$C_{19}H_{36}O_2$	C18:1	296.50
Methy linoleate	$C_{19}H_{34}O_2$	C18:2	294.48
Methy linolenate	$C_{19}H_{32}O_2$	C18:3	292.46

2.4 TRANSESTERIFICATION PROCESS

Transesterification process is the process of converting vegetable & plant oil into biodiesel fuel. Chemically, transesterification means using a triglyceride molecule or a complex fatty acid and neutralizing the free fatty acids then removing the glycerin and will be creating an alcohol ester. The transesterification reaction is represented by the general equation as in the following equation 2.1. Transesterification is one of the reversible reactions and proceeds essentially by mixing with the reactants. The presence of a catalyst (a strong acid or alkali) will accelerate the conversion.



Transesterification of triglycerides with methanol and the presence of the catalyst will produce methyl ester and glycerol. The glycerol layer will be settles down at the bottom of the reaction vessel. In presence of excess alcohol, the forward reaction is first order reaction and the reverse reaction is found to be second order reaction. It was observed that transesterification is faster when catalyzed by alkali (Fatimah, 2008).

2.5 WASTE COOKING OIL

The raw material coming from waste vegetable oils or commonly known as waste cooking oils is one of the alternative sources. Waste cooking oil is easy to get from industries such as domestic usage and restaurant and also cheaper than other oils (refine oils) (Fatimah, 2008). Thus, neat vegetable oil is the best starting material compare to waste cooking oil because of the conversion of triackylglycerides to fatty acid methyl ester is high and the reaction time is relatively short. Waste cooking oil contains higher free fatty acid than neat vegetable oil (Ghadafi, 2008). Physical and chemical properties of waste cooking oil and palm oil can be shown in **Table 2.6**.

Table 2.6: Physical and chemical properties of used frying oil and neat palm oil (Ghadafi, 2008)

Property	UFO*	Neat Palm Oil
Acid value (mg KOH/g)	2.1	< 0.5
Kinematic viscosity at 40 °C (cSt)	35.3	30.2
Fatty acid composition (wt.%)		
Myristic (C14:0)	0.9	1
Palmitic (C16:0)	20.4	42.8
Stearic (C18:0)	4.8	4.5
Oleic (C18:1)	52.9	40.5
Linoleic (C18:2)	13.5	10.1
Linolenic (C18:3)	0.8	0.2
Others	6.7	0.9

* Sample has been pre-treated by filtering and dehydration before analysis.

As can be seen in the table, the waste cooking oil has properties much different from those from the neat oil. The advantages of using waste cooking oil are the low cost and prevention of environment pollution. Waste cooking oil need to be treat before dispose to the environment to prevent pollution. Due to the high cost of disposal, many people dispose waste cooking oil directly to the environment especially in rural area. Then, by recycling waste cooking oil will help to prevent pollution in the environment.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter mainly presents the materials and the experimental procedure of the characterization of cement kiln dust and transesterification process to produce biodiesel from waste cooking oil. There are three methods that have been used to characterize the catalyst which is to measure the pH value using Mettler Toledo pH meter, to know the functional group of the catalyst with Nicolet Avatar 370 DTGS Fourier Transform Infrared Spectrometry (FT-IR) and to measure the change in weight with the relation change in temperature using TGA Q500 Thermogravimetric analyzer (TGA). Biodiesel has been produced using transesterification process using waste cooking oil as a raw material. The presence of biodiesel then was detected by the Agilent Technologies 5975C Gas chromatography with a mass selective detector (GC MS).

3.2 MATERIALS

This section of raw materials was including the raw materials that have been used and the specific place obtained, chemicals and also all the equipment used throughout handling this research.

3.2.1 Raw materials

The raw materials that have been used for this research were cement kiln dust (CKD) and waste cooking oil (WCO). Cement kiln dust was obtained from Pahang Cement Sdn. Bhd. or better known as YTL Cement Berhad and waste cooking oil was picked from the residential college's cafeteria.

3.2.2 Chemical materials

Potassium hydroxide was used for the impregnation step in the catalyst preparation. The potassium hydroxide was supplied by R & M Chemicals to the FKKSA laboratory. The other chemical used was methanol (analytical reagent) also for catalyst preparation and transesterification process. Hexane (analytical grade) was used for dilute the sample preparation for detected the biodiesel compound in the Gas chromatography with a mass selective detector (GC MS). Both were supplied by Fisher Scientific to FKKSA laboratory.

3.2.3 Equipments

The equipment used for this research including Carbolite CWF 1200 muffle furnace, TGA Q500 Thermogravimetric analyzer (TGA), Nicolet Avatar 370 DTGS Fourier Transform Infrared Spectrometry (FT-IR), Mettler Toledo pH meter, Buchi Rotavapor R-200 rotary evaporator, Eppendorf centrifuge 5810 R and Agilent Technologies 5975C gas chromatography with a mass selective detector (GC MS).

3.3 EXPERIMENTAL PROCEDURE

All experimental procedures were being told in detailed in this part. It is including the catalyst preparation, characterization of the catalyst using Mettler Toledo pH meter, Nicolet Avatar 370 DTGS Fourier Transform Infrared Spectrometry (FT-IR), TGA Q500

TGA Q500 Thermogravimetric analysis (TGA), and also the catalyst activation and transesterification process for biodiesel production.

3.3.1 Catalyst Preparation

All the catalyst was prepared by incipient wetness impregnation of CKD with solution of potassium hydroxide (KOH), methanol (CH₃OH) and water (H₂O). For this purpose, the required amount of aqueous KOH, CH₃OH, and H₂O solution was slowly added with the CKD. Four samples have been prepared which are CKD, CKD/KOH, CKD/CH₃OH and CKD/H₂O. Amounts of aqueous solution, such as KOH, CH₃OH, and H₂O, were 10 wt. % of the catalysts prepared (Ilgen and Akin, 2008). The catalysts were dried at 200°C for 1 h. Calcination was performed in a Carbolite CWF 1200 muffled furnace at 650°C for 3 h under static air after crushing the hydrated CKD. Activities of the prepared catalyst were investigated under different aqueous solution of alkaline compounds.

3.3.2 Catalyst characterization

3.3.2.1 pH measurement

The alkalinity of the solid bases was determined by using Mettler Toledo pH meter. About 1g of sample was shaken with an appropriate volume of a methanol solution and distilled water which is about 7.5 ml, and left it a few minutes to equilibrate. The pH reading then was obtained for both using distilled water and methanol and the value of pH reading was determined.

3.3.2.2 Fourier Transform Infrared Spectrometry (FT-IR) analysis

FT-IR spectra of the samples were obtained between 4000 and 500 cm^{-1} on a KBr powder with a Nicolet Avatar 370 DTGS FT-IR spectrometer. A minimum of 64 scans was signal averaged in the 4000-500 cm^{-1} range.

3.3.2.3 Thermogravimetric Analysis

Thermogravimetric analysis (TGA) was performed on a TGA Q500 Thermogravimetric Analyzer. The programmed heating range was from room temperature to 950°C, at a heating rate of 10°C/min under a nitrogen atmosphere (Wang et al., 2011). The measurement was taken using 5 mg of samples.

3.3.3 Catalyst Activation

The methanol and the catalyst were activated for 2 h at 60°C before the transesterification process started. The ratio (v/v) of waste cooking oil to methanol is 1:5 and the weight of catalyst used was 4% from the solvent. The waste cooking oil was filtered until the colour more clear and heated up to 60°C for a few minutes.

3.3.4 Transesterification process

The conversion of waste cooking oil to biodiesel was performed in a 250 ml flat-bottom flask equipped with a magnetic stirrer. Waste cooking oil (WCO) was obtained from a cafeteria in the residential college. The transesterification reaction of WCO and methanol was carried out in liquid phase under atmospheric pressure, at 60°C for 3 h. After the reaction, the solid catalyst was separated by filtration. The liquid was put into a separating funnel and was kept at ambient temperature for 20-30 minutes. After which 2 liquid phases appeared, the upper layer was obtained as a biodiesel and the lower layer was glycerol.

The content in FAME (fatty acid methyl esters) of the upper layer was taken and then centrifuged with Eppendorf centrifuge 5810 R at 5000 rpm for 5-10 minutes to separate the glycerol because it was insoluble in the esters and had a much higher density (Yihuai Li et al., 2010). Then methanol was removed using Buchi Rotavapor R-200 rotary evaporation. For bleaching step which to get clearer colour of FAME, the fuller earth was added and heat up until 50°C and stirred it about 5 minutes. Leaved it for 20 minutes and then centrifuged it again. The product was analyzed by Agilent Technologies 5975C gas chromatography with a mass selective detector (GC MS) to determine the biodiesel yield (fatty acid methyl ester, FAME).

3.3.5 Biodiesel analysis

3.3.5.1 Sample preparation

Before the sample was injected to the GC MS, the sample was prepared by 2% of dilution sample. The dilution of the sample was prepared by adding 20 μL of Fatty acid methyl ester (FAME) and 980 μL of hexane (analytical grade). After the sample was diluted, it must be filtered using 0.2mm syringe filter and filter it into a 1.5 ml vial.

3.3.5.2 Gas chromatography with a mass selective detector (GC MS) analysis

The FAME concentration expressed as the biodiesel purity of the product was determined by gas chromatography with a mass selective detector (GC MS) equipped with a flame ionization detector, a capillary column (Tr-biodiesel (F), Thermo Co., 30 m in length with 0.25 mm i.d. and 0.25 mm film thickness), the programmed column oven, and a programmed temperature injector. The oven temperature program consisted of: start at 120°C (keep 1 min), ramp at 30°C/min to 220°C (keep 1 min), then continue ramp at 10°C/min to 250°C (keep 1 min). The temperature of the programmed temperature injector was 90°C for 0.05 min, programmed to 260°C, at a rate of 10°C/min. Nitrogen was as a

carrier gas with a flow-rate of 2 ml/min (Wang et al., 2011). The purity of FAME can be determined in the percentage area from the peak in the graph obtained and the yield of biodiesel can be calculated as equation 3.1 and 3.2 below.

$$\text{Product yield} = \text{mass}_{\text{fame}} / \text{mass}_{\text{wco}} \times 100\% \quad (3.1)$$

$$\text{Biodiesel yield} = (\text{mass}_{\text{fame}} \times \text{purity of biodiesel}) / (\text{mass}_{\text{wco}}) \quad (3.2)$$

3.4 Summarize Experimental Procedure

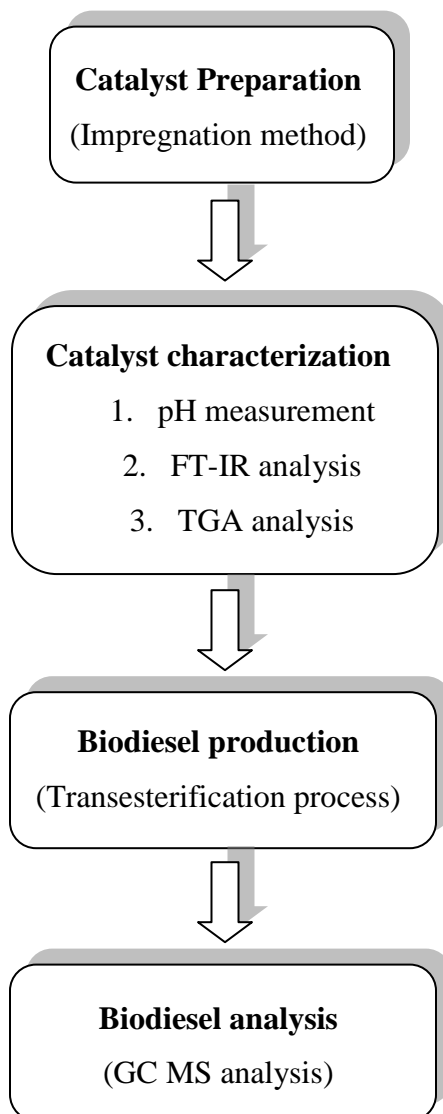


Figure 3.1: Summarize experimental procedure

CHAPTER 4

RESULT AND DISCUSSION

4.1 INTRODUCTION

In this chapter mainly present the characterized of the four sample of catalyst with different alkali solution and the yield of the biodiesel got from the different catalyst impregnation solution. All the experimental data obtained have been evaluated and compared with available or approximately same experimental data from the literature.

4.2 CATALYST CHARACTERIZATION

4.2.1 pH measurement

There are two different controllers that were been used for measured the pH of the catalyst. The first controller is deionized water and the other one is methanol. The pH was measured by Mettler Toledo pH meter to know the value of pH for the entire catalyst sample. From the pH measurement of deionized water and methanol, the pH value get are 6.73 for deionozed water and 6.57 for methanol. From **Table 4.1** the pH values for entire samples were in the range of 9.86 to 11.77. It is shows that the catalyst is in the alkali condition. The strongest alkalinity value is in the sample of CKD/KOH catalyst which is 11.77 and the weakest basicity is 9.86 from the CKD/H₂O catalyst.

According to A.R. Farha (2008), during the transesterification process, the triglyceride is reacted with alcohol in the presence of a catalyst, usually a strong alkaline (NaOH, KOH or sodium silicate). The main reason this process to produce biodiesel, is to find out how much alkaline is needed to ensure a complete transesterification.

Table 4.1: pH of the catalyst

Catalyst/PH	CKD	CKD/KOH	CKD/CH ₃ OH	CKD/H ₂ O
Deionozed water (pH=6.73)	11.25	11.77	10.65	10.25
Methanol(CH ₃ OH) (pH=6.57)	10.32	11.40	10.18	9.86

4.2.2 Fourier Transform Infrared Spectrometry (FT-IR) analysis

The FT-IR analysis of catalyst was performed by using Nicolet Avatar 370 DTGS at FKKSA laboratory. **Figures 4.1** shows the wavelength of CKD, CKD/KOH, CKD/CH₃OH and CKD/H₂O catalyst obtained from the analysis.

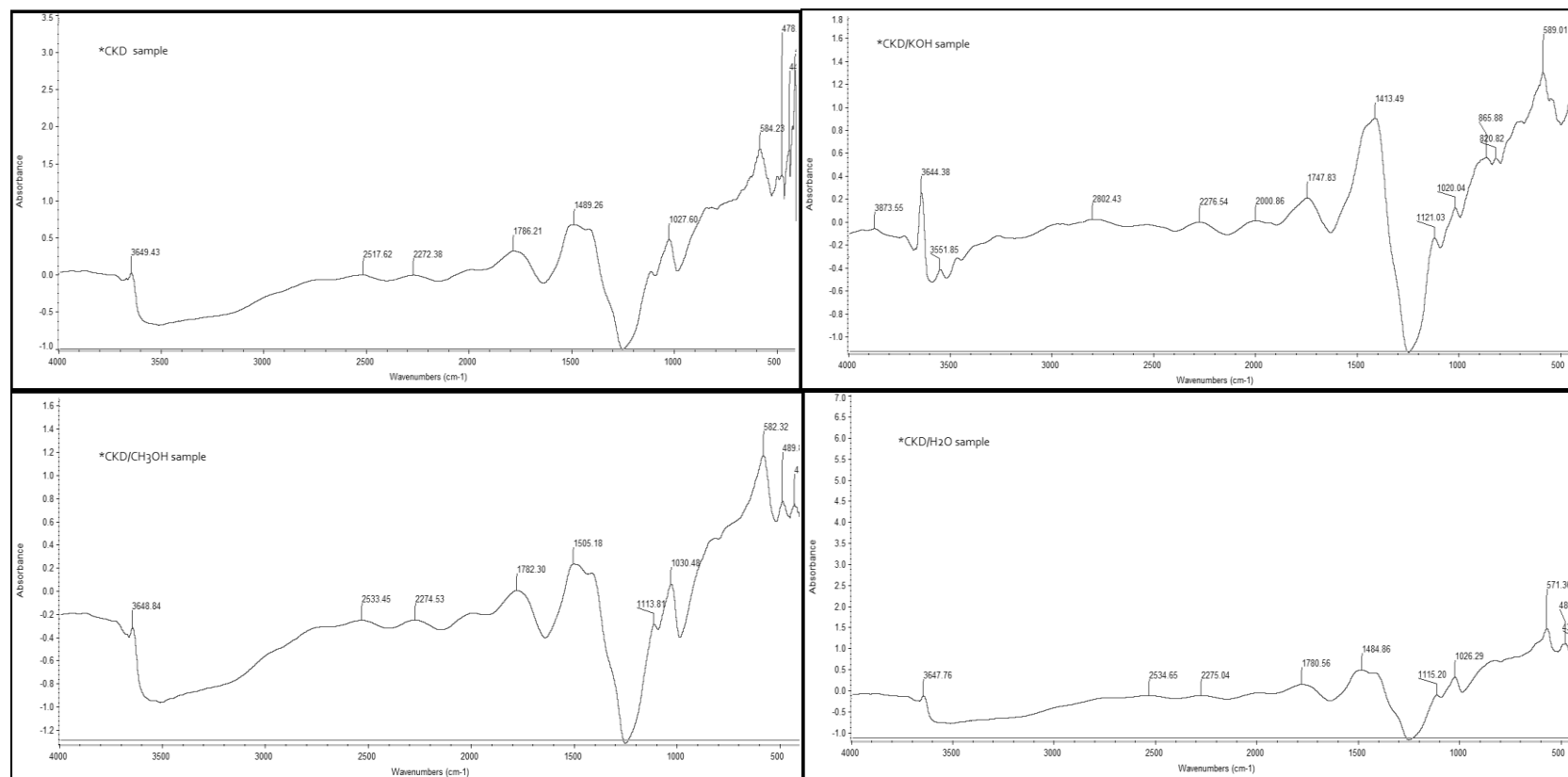


Figure 4.1: FT-IR wavelength from 4000-500 cm⁻¹ a) CKD sample, b) CKD/KOH sample, c) CKD/CH₃OH sample and d) CKD/H₂O sample

Table 4.2: Wave number (cm^{-1}) of dominant peak obtained from absorption spectra

Functional group	CKD	CKD/KOH	CKD/CH ₃ OH	CKD/H ₂ O
Alkanes				
C-H stretch	-	2802.43	-	-
Alkyne				
C \equiv N	2272.38	2276.54	2274.53	2275.04
Anhydride				
O=C-O-C=O	1786.21	-	1782.30	1780.56
Ester				
C=O stretch	-	1747.83	-	-
Carboxylic acid				
C=O stretch	-	1747.83	-	-
O-H absorbs	2517.62	-	2533.45	2534.65
Aliphatic Aldehyde				
C=O stretch	-	1747.83	-	-
Aromatics				
C-C stretch (in-ring)	-	1413.49	-	-
Alcohols				
C-O stretch	1110.0	1121.03	1113.81	1115.20
Alkenes				
=C-H bend	-	820.82-865.88	-	-

The FTIR spectra from **Figure 4.1** reveal several bands: The intensity band at 584, 589, 582 and 571 cm^{-1} for the four catalysts which is characteristic of the metal-oxygen vibrations (possibly Ca-O, Zn-O, Zr-O) (Ezz-Eldin, 2011). The bands at 1020-1027 cm^{-1} can be attributing to SiO_2 and the vibration of bridging oxygen normal to the Si-O-Si plane. The peaks located from 1120-1121 cm^{-1} can be attributed to the C-O bonding of CaCO_3 . The peak at about 1747 cm^{-1} is due to the C=O bonding of CaCO_3 (Chen et al., 2011). In addition the infrared spectra for the other bands at about; 2517.62-2534.65 cm^{-1} which are generally correlated to the stretching and deforming modes for (OH) groups, molecular water or silanol groups (Ezz-Eldin, 2011). The others band is shown in the **Table 4.2** for the dominant peak obtained from absorption spectra.

4.2.3 Thermogravimetric Analysis

The percentage weight loss of the catalyst was analyzed using Thermogravimetric analyzer (TGA Q500) at FKKSA laboratory. From the graph that obtained, just CKD/KOH catalyst has lost their weight after heating it to 950°C. The other catalyst showed that at 600 °C and above, the percentage weight of the catalyst was increased. It can be said that the catalyst was not decomposed when heating in the high temperature for the time being.

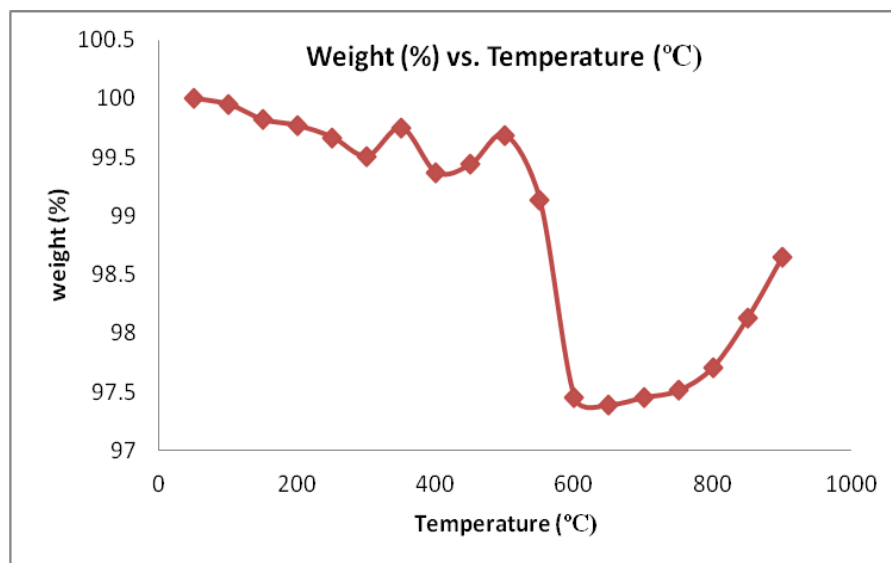


Figure 4.2: CKD catalyst

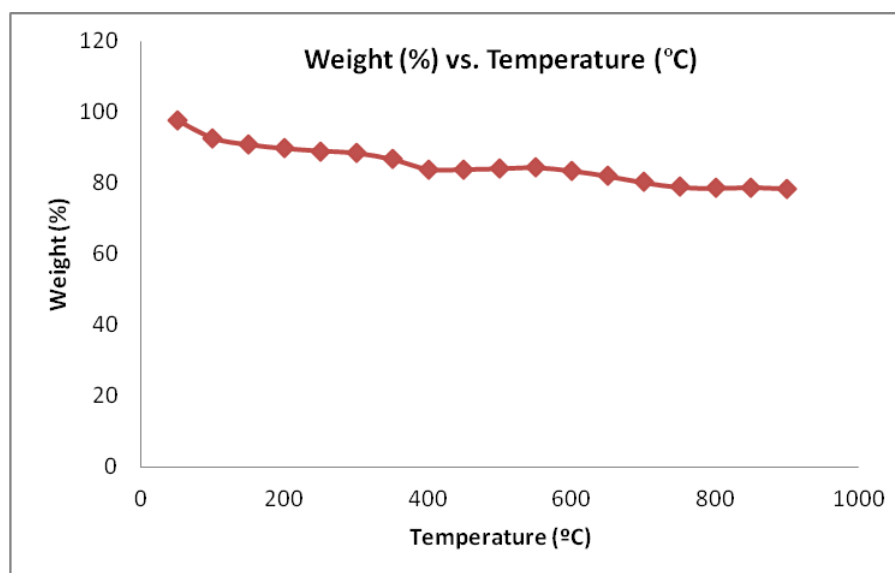


Figure 4.3: CKD/KOH catalyst

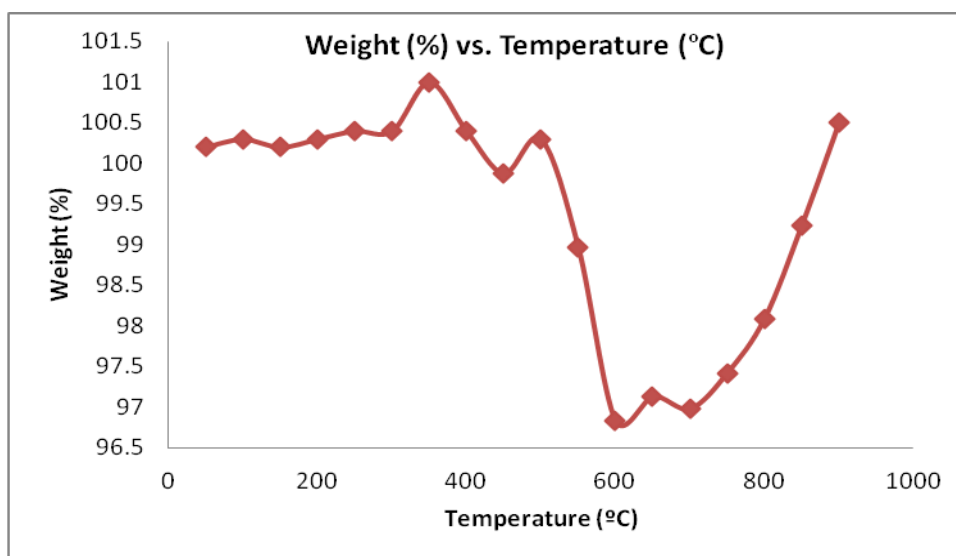


Figure 4.4: CKD/CH₃OH catalyst

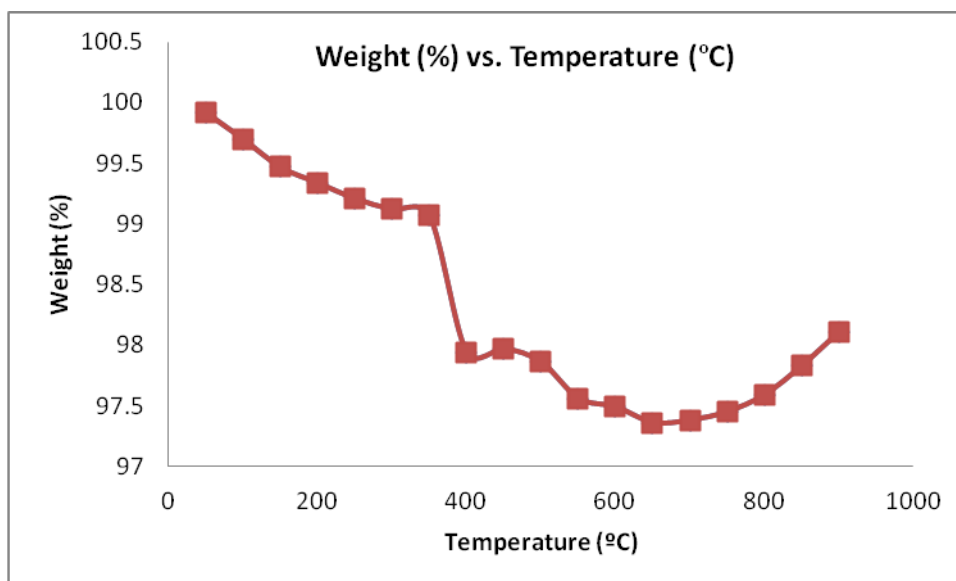


Figure 4.5: CKD/H₂O catalyst

Ignou, 2011 said, there are some possible sources of error in TGA analysis and they can lead to inaccuracies in the recorded temperature and mass data. Some of the errors may be corrected by placing the thermo balance at proper place and handling it with great care.

The other is because the Buoyance effect. If a thermally inert crucible is heated when empty there is usually an apparent weight change as temperature increases. This is due to effect of change in buoyancy of the gas in the sample environment with the temperature. Condensation of the sample will also affect the mass of the sample and consequently the shape of TG curve .This can be avoided by maintaining a dynamic atmosphere around the sample in the furnace. Besides, random fluctuation of balance mechanism, reaction between sample and container, convection effect from furnace, turbulence effect from gas flow also can affect the graph of TGA.

Nonanoic acid	0	0	0	0	0	0	10.607	0.47	0	0
ME – Tridecanoic acid	0	0	12.893	0.47	12.946	0.75	0	0	0	0
ME - Pentadecanoic acid	0	0	0	0	0	0	13.929	0.04	0	0
ME – 7-Hexadecenoic acid,9-Hexadecenoic acid, 11-Hexadecenoic acid	0	0	14.816	1.59	0	0	0	0	14.816, 14.869, 14.944	0.60, 0.60, 0.67
ME - 7-Hexadecenoic acid, 9-Octadecenoic acid	0	0	0	0	0	0	14.826	3.19	0	0
ME - Hexadecanoic acid, pentadecanoic acid	0	0	15.435	29.85	15.585	33.26	15.553	30.03	15.585	31.68
ME – Heptadecanoic acid, Hexadecanoic acid	0	0	0	0	0	0	16.578	0.12	0	0
ME – 11,14-Octadecadienoic acid, 9,12-Octadecadienoic acid	0	0	17.646	1.73	0	0	17.646	1.51	0	0
ME – 9-Octadecenoic acid, 8-Octadecenoic acid	0	0	18.052, 18.319	34.70, 21.41	18.362	55.62	18.309	53.09	0	0

10-Octadecenoic acid										
ME- 9-Octadecenoic acid	0	0	0	0	0	0	19.719	0.23	0	0
ME - Eicosanoic acid	0	0	19.826	0.27	19.847	0.51	19.847	0.32	19.847	0.46
ME - 4,7,10,13,16,19- Docosahexaenoic acid, 5,8,11,14,17- Eicosapentaenoic acid	0	0	0	0	0	0	20.734	0.23	20.723	0.20
ME - 9,12,15- Octadecatrienoic acid	0	0	0	0	20.798	0.18	0	0	0	0
TOTAL Methyl Ester (% Area)				97.55		97.85		96.89		96.6

*ME - Methyl ester

From GC MS analysis, the total percentage area that obtained from the CKD catalyst is 97.55%, CKD/KOH is 97.85%, CKD/CH₃OH equal to 96.89% and CKD/H₂O is 96.6%. The result from **Table 4.2** shows, the highest content of fatty acid is 9-Octadecenoic acid, 8-Octadecenoic acid, which is about 34.70%-53.09% and the second highest is hexadecanoic acid, pentadecanoic acid which is 29.85%-33.26%. It is shows that the percentage of fatty acid produce is likely same as mentioned from Lam et al. (2010) which is the composition of palmitic is 42.8% and oleic fatty acid is 40.5% for the biodiesel production from palm oil.

The value of total percentage area is same with the purity of the biodiesel produce. To get the yield of the biodiesel, equation 3.1 and 3.2 in the literature review was used.

$$\text{Product yield} = \text{mass}_{\text{fame}} / \text{mass}_{\text{wco}} \times 100\% \quad (3.1)$$

$$\text{Biodiesel yield} = (\text{mass}_{\text{fame}} \times \text{purity of biodiesel}) / (\text{mass}_{\text{wco}}) \quad (3.2)$$

From the calculation, the yield of biodiesel for CKD catalyst is about 89.07%, CKD/KOH is about 89.34% , 88.46% yield of biodiesel for CKD/CH₃OH and 88.2% for CKD/H₂O catalyst. Therefore, the highest yield produce from the CKD/KOH catalyst that contained the highest alkalinity value compared to the other catalyst. It also can be shown that CKD is not impregnate with the other catalyst also get highest yield of biodiesel production. It is because when the CKD was calcinated in the high temperature it can become CaO. It can be said that, CKD's own have the highest basicity of the alkali and have a good condition to be an alkali catalyst for transesterification process in the biodiesel production.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

In this study, from the pH analysis, the highest alkalinity value is CKD/KOH catalyst and the lower alkalinity is CKD/H₂O catalyst. It is maybe due to the concentration of the CKD/H₂O is lower when it impregnate with the water. The functional group that can be obtained from the FT-IR analysis, there are nine functional group that can be detected like ester, alkayne, alkanes, carboxylic acid, anhydrate, aliphatic aldehyde, aromatics, alcohol and alkenes. From the percentage of weight loss analysis due to the change of temperature with TGA Q500, the weight loss and decomposition of catalyst can be seen in the CKD/KOH catalyst.

From the purity obtained in the GC MS data, CKD/KOH gives the highest purity which is 97.85%, CKD gives 97.55%, CKD/CH₃OH got 96.89% and CKD/H₂O reading is 96.6%. CKD/KOH catalyst gives the highest yield of biodiesel production which is 89.34%, followed by CKD which is 89.07%, CKD/CH₃OH is 88.46% and 88.2% for CKD/H₂O catalyst. Therefore, waste CKD catalyst was successfully tested in the transesterification reaction of waste cooking oil. The catalyst can be produce with a low cost of material and high efficiency of production.

5.2 RECOMMENDATIONS

From this study, there are a lot of improvements that can be used to get the better result. In the part of catalyst characterization such as using Powder X-ray diffractometer (XRD) to know the crystallization phase and compound, characterize the surface area, total pore volume, pore diameter and pore size using BET method, morphology and size using scanning electron microscopy (SEM), and also can test the density of the catalyst.

There are some analyses that can be improved like in the pH analysis with the Hammet Indicator test to know the basicity of the catalyst. In the TGA analysis, the result obtained is not so accurate, because just CKD/KOH has lost their weight, but for the others catalyst not. Therefore, maybe it can be programmed the heating under an oxygen atmosphere compared using nitrogen.

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APPENDICES

APPENDIX A

Termogravimetric analysis graph

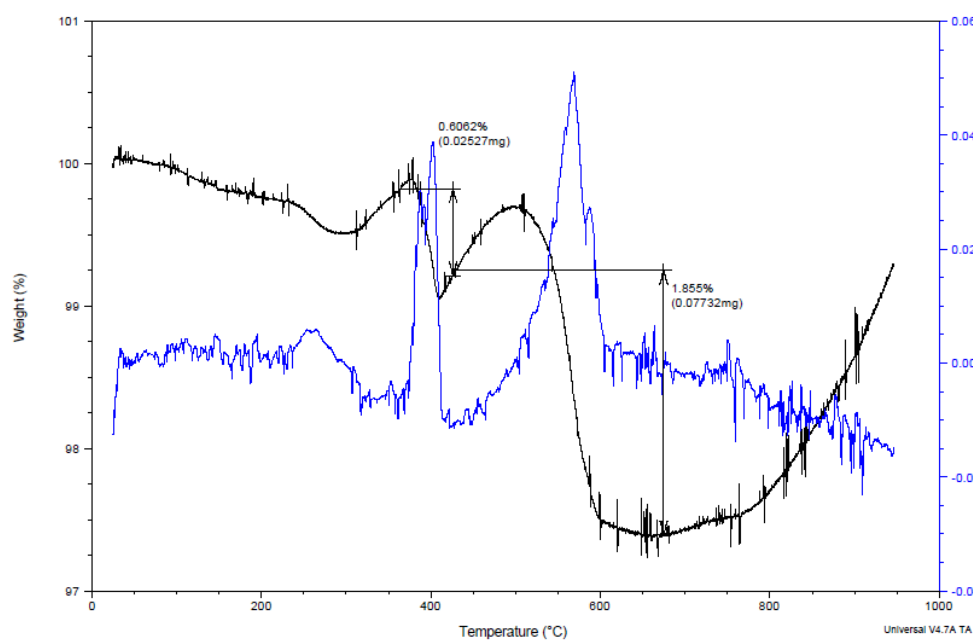


Figure 1: CKD catalyst

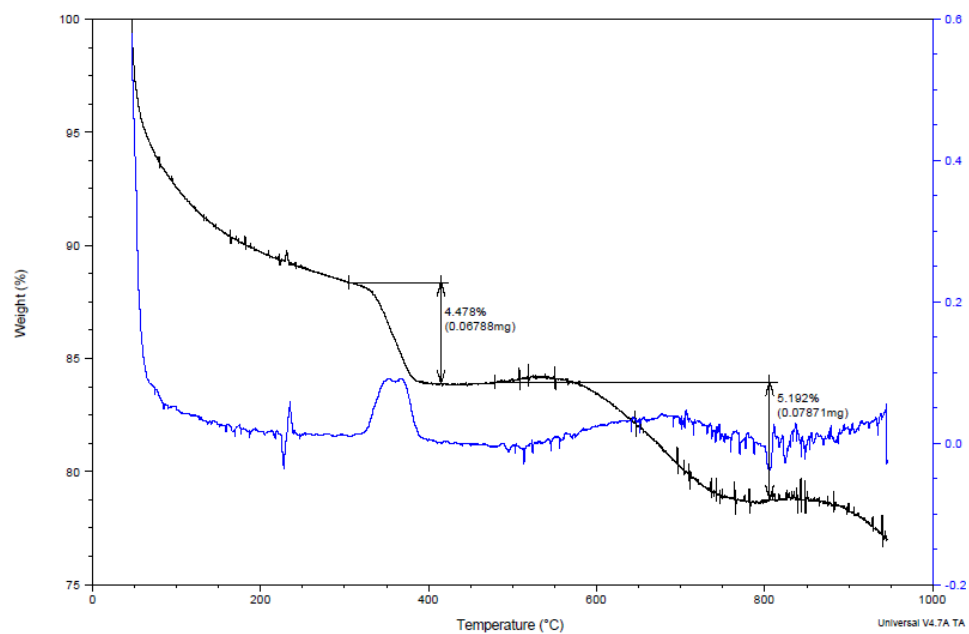
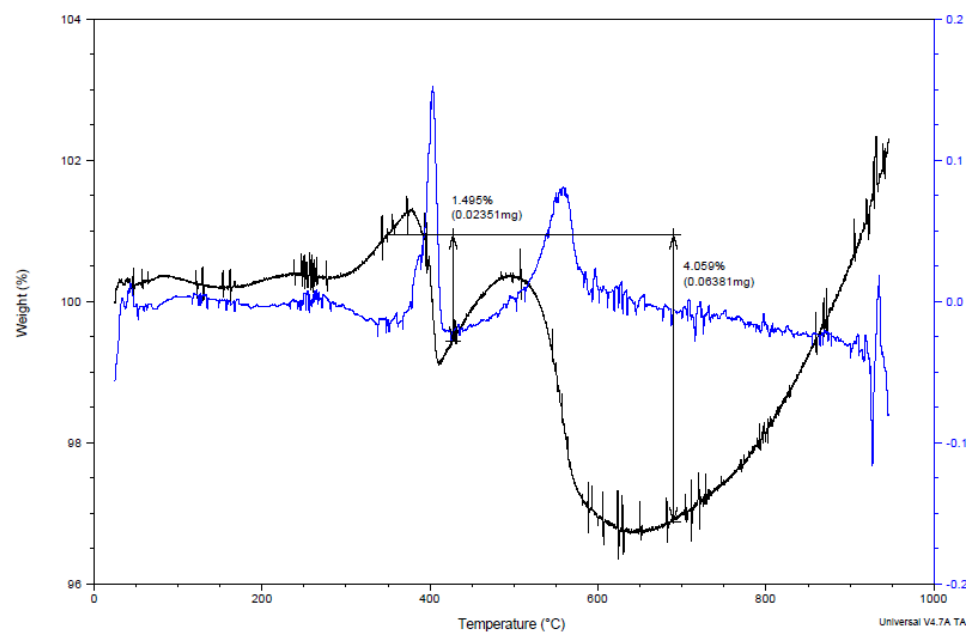


Figure 2: CKD/KOH catalyst

Figure 3: CKD/CH₃OH catalyst

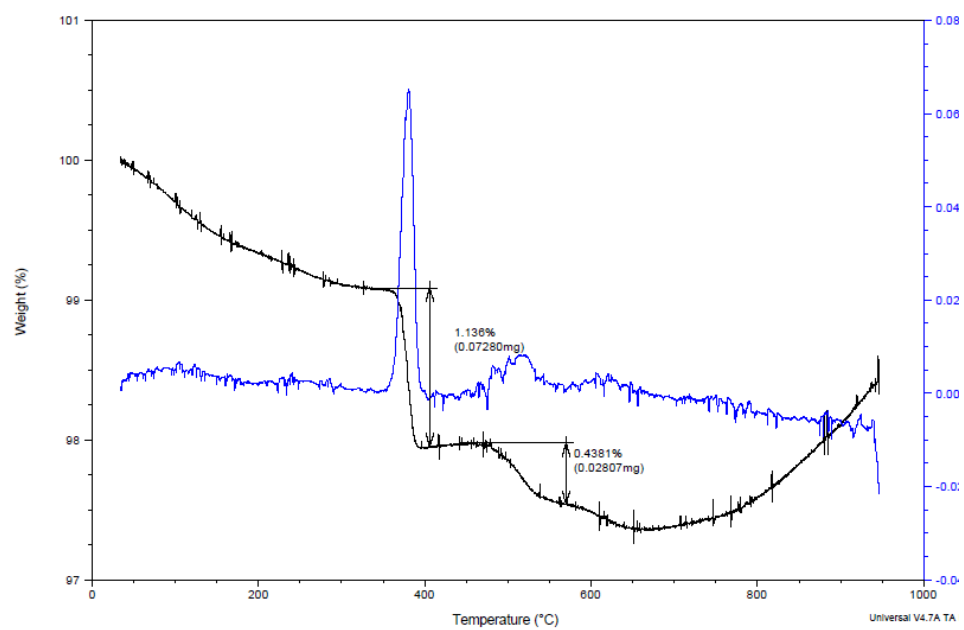
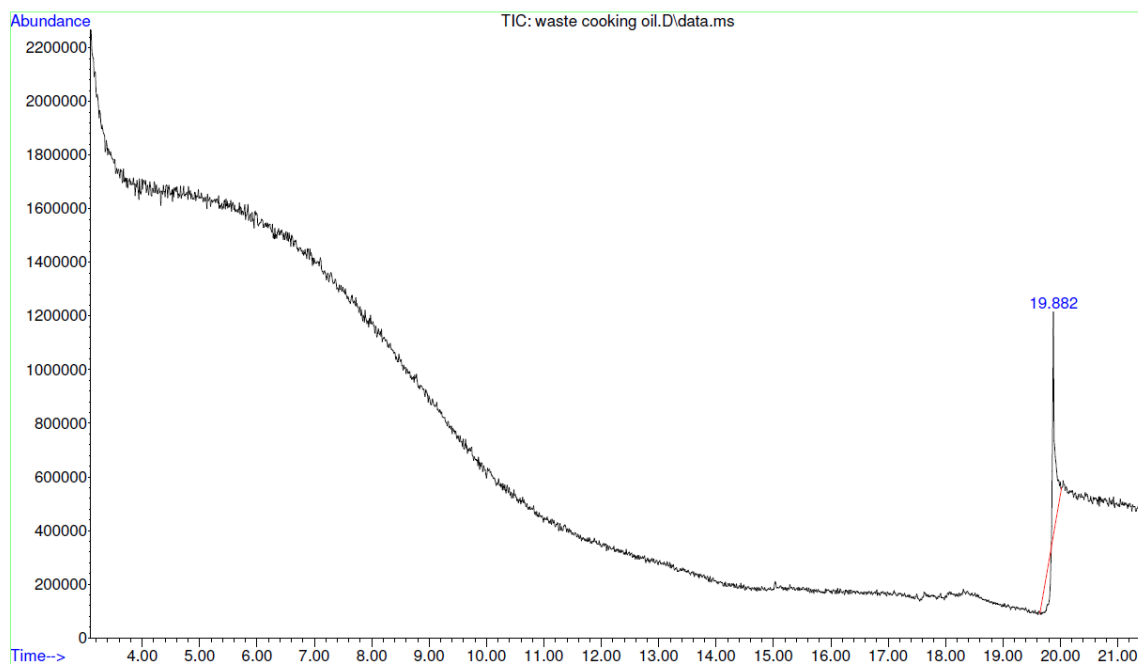


Figure 4: CKD/H₂O catalyst

APPENDIX B**Gas chromatography with a mass selective detector (GC MS) graph****Figure 5: Waste cooking oil peak**

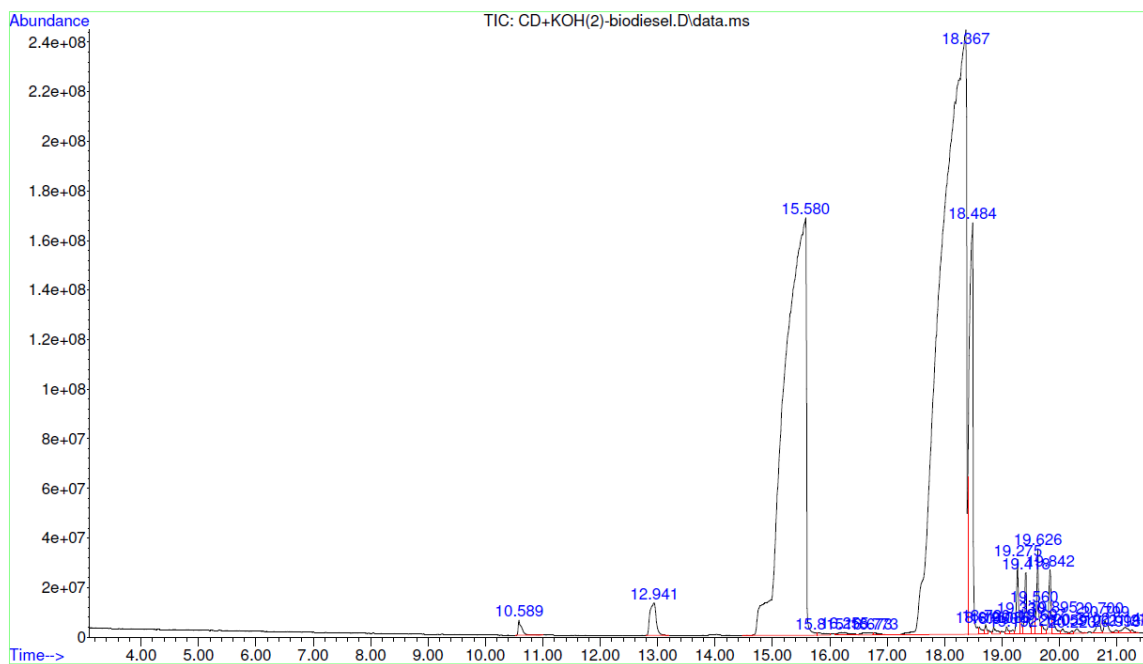


Figure 5: Biodiesel yield for CKD/KOH catalyst

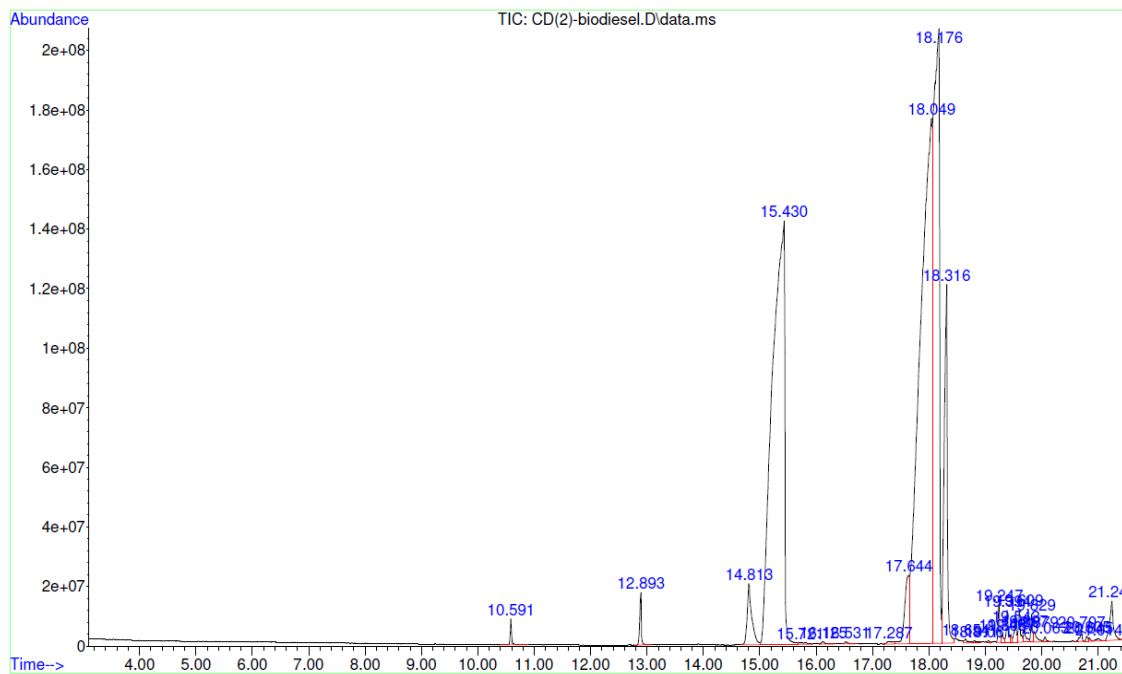


Figure 6: Biodiesel yield for CKD catalyst

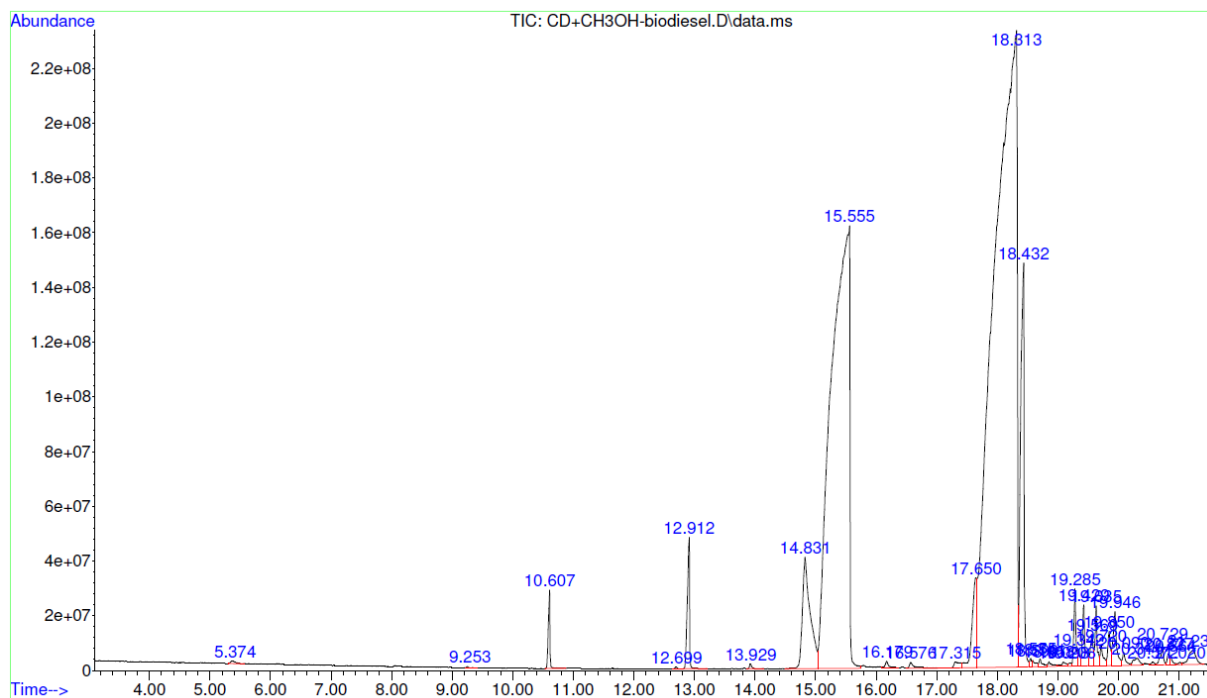


Figure 7: Biodiesel yield for CKD/CH₃OH catalyst

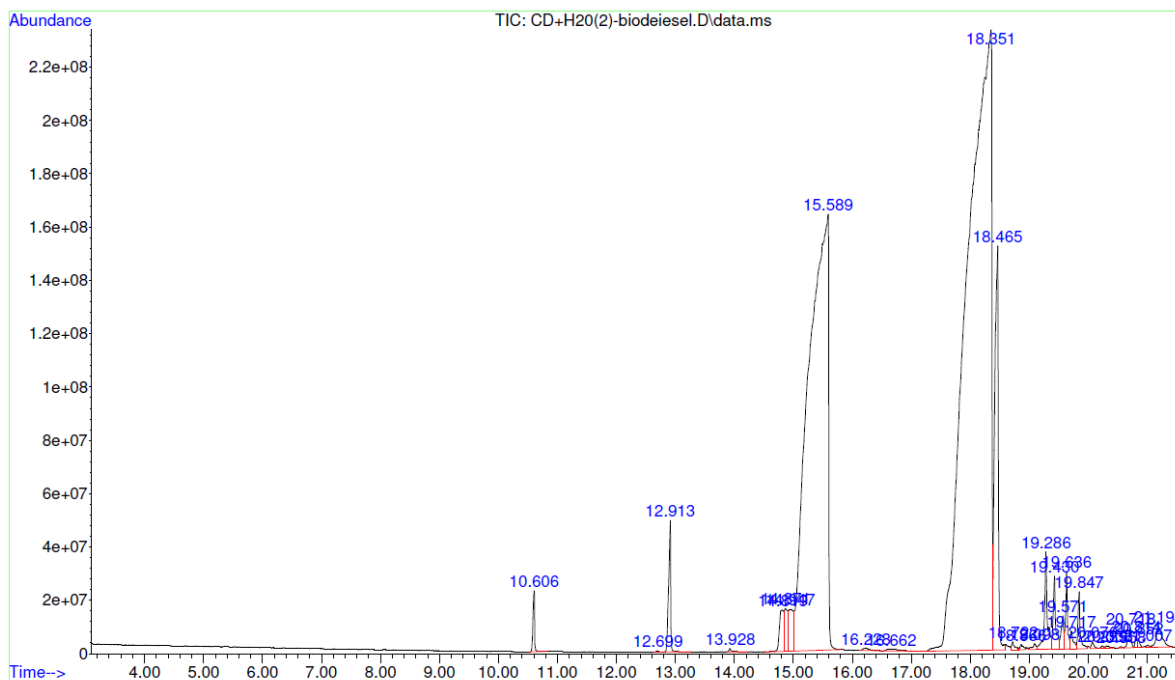


Figure 8: Biodiesel yield for CKD/H₂O catalyst

APPENDIX C

Datasheets result from GC MS

1. Waste Cooking Oil

Library Search Report

Data Path : D:\Data\psm2011\waste cooking oil 4jan12\
Data File : waste cooking oil.D
Acq On : 4 Jan 2012 9:34
Operator : FIZA4JAN
Sample : waste cooking oil
Misc :
ALS Vial : 1 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e

PK#	RT	Area%	Library/ID	Ref#	CAS#	Qual
1	19.879	100.00	C:\Database\NIST05a.L			
			2,5-Isoxazolidinedicarboxylic acid	59171	015166-62-8	42
			, 2-ethyl 5-methyl ester			
			Pentadecylamine	76642	002570-26-5	9
			1-Heptadecanamine	95504	004200-95-7	9

2. Biodiesel from CKD

Library Search Report

Data Path : D:\Data\psm2011\waste cooking oil 4jan12\
 Data File : CD(2)-biodiesel.D
 Acq On : 4 Jan 2012 10:01
 Operator : FIZA4JAN
 Sample : CD(2)-biodiesel
 Misc :
 ALS Vial : 3 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
 Integration Events: ChemStation Integrator - autoint1.e

Pk#	RT	Area%	Library/ID	Ref#	CAS#	Qual
1	10.586	0.19	C:\Database\NIST05a.L Dodecanoic acid, methyl ester	67167	000111-82-0	95
			Dodecanoic acid, methyl ester	67169	000111-82-0	87
			Dodecanoic acid, methyl ester	67168	000111-82-0	80
2	12.893	0.47	C:\Database\NIST05a.L Methyl tetradecanoate	86750	000124-10-7	97
			Tridecanoic acid, 12-methyl-, methyl ester	86778	005129-58-8	94
			Methyl tetradecanoate	86752	000124-10-7	86
3	14.816	1.59	C:\Database\NIST05a.L 7-Hexadecenoic acid, methyl ester, (Z)-	104151	056875-67-3	99
			9-Hexadecenoic acid, methyl ester, (Z)-	104152	001120-25-8	99
			11-Hexadecenoic acid, methyl ester	104135	055000-42-5	91
4	15.435	29.85	C:\Database\NIST05a.L Hexadecanoic acid, methyl ester	105639	000112-39-0	86
			Pentadecanoic acid, 14-methyl-, methyl ester	105659	005129-60-2	81
			Hexadecanoic acid, methyl ester	105643	000112-39-0	55
5	15.724	0.08	C:\Database\NIST05a.L 1-Nonanamine	19630	000112-20-9	9
			2,4(1H,3H)-Pyrimidinedione, 6-chloro-5-nitro-	50640	006630-30-4	7
			1-Butanamine, 3-methyl-	1888	000107-85-7	5
6	16.130	0.06	C:\Database\NIST05a.L 2H-Pyran, 2-(3-butynyloxy)tetrahydro-	26517	040365-61-5	9
			9-Octadecene, 1,1-dimethoxy-, (Z)-	132340	015677-71-1	9
			Cyclopentanol, 2-(aminomethyl)-, trans-	7660	040482-02-8	4
7	16.536	0.04	C:\Database\NIST05a.L 1-Hexadecanamine	86072	000143-27-1	9
			1,10-Diaminodecane	37678	000646-25-3	9
			Acetamide, N-ethyl-	1849	000625-50-3	7
8	17.283	0.09	C:\Database\NIST05a.L Phenylethanolamine	16140	007568-93-6	12
			Phenylethanolamine	16144	007568-93-6	9
			1,10-Diaminodecane	37678	000646-25-3	9
9	17.646	1.73	C:\Database\NIST05a.L 11,14-Octadecadienoic acid, methyl ester	121099	056554-61-1	99
			9,12-Octadecadienoic acid (Z,Z)-, methyl ester	121107	000112-63-0	99
			9,12-Octadecadienoic acid (Z,Z)-, methyl ester	121105	000112-63-0	99
10	18.052	34.70	C:\Database\NIST05a.L 9-Octadecenoic acid, methyl ester, (E)-	122326	001937-62-8	99
			9-Octadecenoic acid, methyl ester	122299	002462-84-2	99

Library Search Report

Data Path : D:\Data\psm2011\waste cooking oil 4jan12\
 Data File : CD(2)-biodiesel.D
 Acq On : 4 Jan 2012 10:01
 Operator : FIZA4JAN
 Sample : CD(2)-biodiesel
 Misc :
 ALS Vial : 3 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
 Integration Events: ChemStation Integrator - autoint1.e

PK#	RT	Area%	Library/ID	Ref#	CAS#	Qual
			8-Octadecenoic acid, methyl ester	122297	002345-29-1	99
11	18.181	21.41	C:\Database\NIST05a.L 9-Octadecenoic acid, methyl ester, (E)-	122326	001937-62-8	99
			9-Octadecenoic acid, methyl ester	122299	002462-84-2	99
			8-Octadecenoic acid, methyl ester	122297	002345-29-1	99
12	18.319	6.40	C:\Database\NIST05a.L Octadecanoic acid, methyl ester	123709	000112-61-8	96
			Octadecanoic acid, methyl ester	123708	000112-61-8	93
			Heptadecanoic acid, 15-methyl-, me thyl ester	123730	054833-55-5	91
13	18.651	0.08	C:\Database\NIST05a.L No matches found			
14	18.811	0.05	C:\Database\NIST05a.L 9-Octadecene, 1,1-dimethoxy-, (Z)-	132340	015677-71-1	7
			1,5-Dichloro-2,3-dinitrobenzene	82706	028689-08-9	5
			Cyclopentanol, 2-(aminomethyl)-, t rans-	7660	040482-02-8	3
15	19.056	0.02	C:\Database\NIST05a.L 1-Octadecanamine	104870	000124-30-1	9
			1,10-Diaminodecane	37678	000646-25-3	9
			Acetamide, N-ethyl-	1846	000625-50-3	9
16	19.249	0.36	C:\Database\NIST05a.L 5,8,11,14-Eicosatetraenoic acid, e thyl ester, (all-Z)-	143836	001808-26-0	93
			Methyl eicosa-5,8,11,14,17-pentaen oate	134744	001191-65-7	86
			Arachidonic acid	127424	000506-32-1	72
17	19.313	0.08	C:\Database\NIST05a.L 3,8-Dioxatricyclo[5.1.0.0(2,4)]oct ane, 4-ethenyl-	16818	053966-43-1	17
			2,4-Cyclopentadiene-1-ethanamine	5467	138816-65-6	9
			Cyclododecanol, 1-aminomethyl-	66564	000832-29-1	9
18	19.398	0.34	C:\Database\NIST05a.L 9,12,15-Octadecatrienoic acid, met hyl ester, (Z,Z,Z)-	119876	000301-00-8	89
			7,10,13-Eicosatrienoic acid, methy l ester	137079	030223-51-9	87
			7,10,13-Eicosatrienoic acid, methy l ester	137077	030223-51-9	83
19	19.452	0.08	C:\Database\NIST05a.L 1,4-Cyclohexanedimethanamine	19170	002549-93-1	33
			9-Octadecene, 1,1-dimethoxy-, (Z)-	132340	015677-71-1	16
			Ethanol, 2-bromo-	9920	000540-51-2	10
20	19.548	0.24	C:\Database\NIST05a.L 11,14-Eicosadienoic acid, methyl e ster	138089	002463-02-7	98
			11,13-Eicosadienoic acid, methyl e ster	138090	056599-57-6	97

Library Search Report

Data Path : D:\Data\psm2011\waste cooking oil 4jan12\
 Data File : CD(2)-biodiesel.D
 Acq On : 4 Jan 2012 10:01
 Operator : FIZA4JAN
 Sample : CD(2)-biodiesel
 Misc :
 ALS Vial : 3 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
 Integration Events: ChemStation Integrator - autoint1.e

Pk#	RT	Area%	Library/ID	Ref#	CAS#	Qual
			10,13-Eicosadienoic acid, methyl ester	138091	030223-50-8	95
21	19.612	0.36	C:\Database\NIST05a.L 11-Eicosenoic acid, methyl ester	139171	003946-08-5	99
			Cyclopentadecanone, 2-hydroxy-	85349	004727-18-8	43
			2-Cyclopenten-1-one, 2-pentyl-	24112	025564-22-1	20
22	19.697	0.18	C:\Database\NIST05a.L Acetamide, N-ethyl-	1846	000625-50-3	9
			N-(2,2-Dimethyl-propyl)-propionamide	19606	118764-26-4	9
			Acetamide, N-ethyl-	1850	000625-50-3	9
23	19.826	0.27	C:\Database\NIST05a.L Eicosanoic acid, methyl ester	140312	001120-28-1	99
			Eicosanoic acid, methyl ester	140314	001120-28-1	98
			Eicosanoic acid, methyl ester	140313	001120-28-1	98
24	19.879	0.18	C:\Database\NIST05a.L Formamide, N,N'-1,3-propanediylbis	12772	016419-41-3	28
			Pentadecylamine	76642	002570-26-5	11
			Tridecylamine	56580	002869-34-3	10
25	20.061	0.06	C:\Database\NIST05a.L 3-Bromo-3-buten-1-ol	23056	076334-36-6	9
			Octopamine	25339	000104-14-3	7
			2,3-Dimethoxyphenethylamine	43744	003213-29-4	4
26	20.701	0.13	C:\Database\NIST05a.L dl-2-Amino-1-phenylethanol	16178	001936-63-6	23
			Bicyclo[3.1.1]hept-3-ene-spiro-2,4	52732	1000149-76-2	14
			'-(1',3'-dioxane), 7,7-dimethyl-			
			4,9-Decadienoic acid, 2-nitro-, ethyl ester	85828	029085-46-9	10
27	20.808	0.05	C:\Database\NIST05a.L 3,8-Dioxatricyclo[5.1.0.0(2,4)]octane, 4-ethenyl-	16818	053966-43-1	27
			4,9-Decadienoic acid, 2-nitro-, ethyl ester	85828	029085-46-9	12
			Bicyclo[3.1.1]hept-3-ene-spiro-2,4	52732	1000149-76-2	9
			'-(1',3'-dioxane), 7,7-dimethyl-			
28	20.840	0.04	C:\Database\NIST05a.L Bicyclo[3.1.1]hept-3-ene-spiro-2,4	52732	1000149-76-2	43
			'-(1',3'-dioxane), 7,7-dimethyl-			
			3-Methoxyphenethylamine	23932	002039-67-0	9
			2-(2-Methoxyphenyl)ethylamine	23948	002045-79-6	7
29	21.011	0.05	C:\Database\NIST05a.L Ethanol, 2-bromo-	9919	000540-51-2	7
			Cyclododecanol, 1-aminomethyl-	66564	000832-29-1	7
			Oleylamine	103449	000112-90-3	7
30	21.246	0.81	C:\Database\NIST05a.L Hexadecanoic acid, 2,3-dihydroxypropyl ester, (+/-)-	142406	019670-51-0	58
			Hexadecanoic acid, 1-(hydroxymethyl)-	187613	000761-35-3	53

Library Search Report

Data Path : D:\Data\psm2011\waste cooking oil 4jan12\
Data File : CD(2)-biodiesel.D
Acq On : 4 Jan 2012 10:01
Operator : FIZA4JAN
Sample : CD(2)-biodiesel
Misc :
ALS Vial : 3 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e

Pk#	RT	Area%	Library/ID	Ref#	CAS#	Qual
			1)-1,2-ethanediyl ester			
			15-Hydroxypentadecanoic acid	97426	004617-33-8	41

3. Biodiesel from CKD/KOH

Library Search Report

Data Path : D:\Data\psm2011\waste cooking oil 4jan12\
 Data File : CD+KOH(2)-biodiesel.D
 Acq On : 4 Jan 2012 10:56
 Operator : FIZA4JAN
 Sample : CD+KOH(2)-biodiesel
 Misc :
 ALS Vial : 5 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
 Integration Events: ChemStation Integrator - autoint1.e

Pk#	RT	Area%	Library/ID	Ref#	CAS#	Qual
1	10.586	0.20	C:\Database\NIST05a.L Dodecanoic acid, methyl ester	67167	000111-82-0	96
			Dodecanoic acid, methyl ester	67169	000111-82-0	90
			Methyl tetradecanoate	86751	000124-10-7	72
2	12.946	0.75	C:\Database\NIST05a.L Methyl tetradecanoate	86750	000124-10-7	97
			Tridecanoic acid, 12-methyl-, methyl ester	86778	005129-58-8	93
			Methyl tetradecanoate	86752	000124-10-7	81
3	15.585	33.26	C:\Database\NIST05a.L Hexadecanoic acid, methyl ester	105639	000112-39-0	84
			Pentadecanoic acid, 14-methyl-, methyl ester	105659	005129-60-2	60
			Pentadecanoic acid, 13-methyl-, methyl ester	105660	005487-50-3	49
4	15.820	0.08	C:\Database\NIST05a.L 2,4(1H,3H)-Pyrimidinedione, 6-chloro-5-nitro-	50640	006630-30-4	9
			1-Dodecanamine	46744	000124-22-1	9
			Oleylamine	103449	000112-90-3	9
5	16.258	0.12	C:\Database\NIST05a.L 2-(3-Methylguanidino)ethanol	8202	1000242-22-5	4
			9-Octadecene, 1,1-dimethoxy-, (Z)-	132340	015677-71-1	4
			1,5-Dichloro-2,3-dinitrobenzene	82706	028689-08-9	4
6	16.674	0.08	C:\Database\NIST05a.L 1-Butanamine, 3-methyl-	1880	000107-85-7	9
			Acetamide, N-ethyl-	1846	000625-50-3	9
			Pentadecylamine	76642	002570-26-5	9
7	16.771	0.05	C:\Database\NIST05a.L Acetamide, N-ethyl-	1846	000625-50-3	9
			1,10-Diaminodecane	37678	000646-25-3	9
			Heptanedioic acid, dimethyl ester	49052	001732-08-7	9
8	18.362	55.62	C:\Database\NIST05a.L 9-Octadecenoic acid, methyl ester, (E)-	122326	001937-62-8	99
			8-Octadecenoic acid, methyl ester	122297	002345-29-1	99
			9-Octadecenoic acid, methyl ester, (E)-	122329	001937-62-8	99
9	18.480	5.96	C:\Database\NIST05a.L Heptadecanoic acid, 14-methyl-, methyl ester, (+/-)-	123733	057274-45-0	93
			Heptadecanoic acid, 14-methyl-, methyl ester	123731	002490-23-5	93
			Octadecanoic acid, methyl ester	123709	000112-61-8	76
10	18.608	0.08	C:\Database\NIST05a.L Ethanol, 2-bromo-	9919	000540-51-2	27
			1,4-Cyclohexanedimethanamine	19170	002549-93-1	16
			1,4-Cyclohexanedimethanamine	19168	002549-93-1	16
11	18.725	0.07	C:\Database\NIST05a.L			

Library Search Report

Data Path : D:\Data\psm2011\waste cooking oil 4jan12\
 Data File : CD+KOH(2)-biodiesel.D
 Acq On : 4 Jan 2012 10:56
 Operator : FIZA4JAN
 Sample : CD+KOH(2)-biodiesel
 Misc :
 ALS Vial : 5 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
 Integration Events: ChemStation Integrator - autoint1.e

Pk#	RT	Area%	Library/ID	Ref#	CAS#	Qual
			1,4-Cyclohexanedimethanamine	19170	002549-93-1	25
			3,8-Dioxatricyclo[5.1.0.0(2,4)]octane, 4-ethenyl-	16818	053966-43-1	17
			9-Octadecene, 1,1-dimethoxy-, (Z)-	132340	015677-71-1	16
12	18.864	0.09	C:\Database\NIST05a.L			
			Ethanol, 2-bromo-	9920	000540-51-2	9
			Ethanol, 2-bromo-	9919	000540-51-2	9
			1-Tetradecanamine	66606	002016-42-4	9
13	19.089	0.06	C:\Database\NIST05a.L			
			Urea, octadecyl-	132227	002158-08-9	43
			1,10-Diaminodecane	37678	000646-25-3	10
			N-(2-Chloroethyl)-N'-methylurea	15455	074378-14-6	10
14	19.195	0.06	C:\Database\NIST05a.L			
			1-Heptadecanamine	95504	004200-95-7	9
			1,7-Diaminoheptane	13169	000646-19-5	9
			Ethanol, 2-bromo-	9919	000540-51-2	9
15	19.270	0.51	C:\Database\NIST05a.L			
			5,8,11,14-Eicosatetraenoic acid, ethyl ester, (all-Z)-	143836	001808-26-0	94
			Arachidonic acid	127424	000506-32-1	83
			1,3-Cyclododecadiene, (E,Z)-	32190	001129-92-6	80
16	19.334	0.10	C:\Database\NIST05a.L			
			4,9-Decadienoic acid, 2-nitro-, ethyl ester	85828	029085-46-9	27
			Benzeneethanamine, .beta.-methyl-	15120	000582-22-9	25
			3,8-Dioxatricyclo[5.1.0.0(2,4)]octane, 4-ethenyl-	16818	053966-43-1	12
17	19.420	0.56	C:\Database\NIST05a.L			
			7,10,13-Eicosatrienoic acid, methyl ester	137079	030223-51-9	94
			Tricyclo[5.3.0.0(3,9)]decane	15226	053130-27-1	87
			9,12,15-Octadecatrienoic acid, methyl ester, (Z,Z,Z)-	119876	000301-00-8	83
18	19.559	0.22	C:\Database\NIST05a.L			
			11,14-Eicosadienoic acid, methyl ester	138089	002463-02-7	99
			11,13-Eicosadienoic acid, methyl ester	138090	056599-57-6	99
			10,13-Eicosadienoic acid, methyl ester	138091	030223-50-8	95
19	19.623	0.59	C:\Database\NIST05a.L			
			11-Eicosenoic acid, methyl ester	139171	003946-08-5	99
			Cyclopentadecanone, 2-hydroxy-	85349	004727-18-8	53
			Cyclopropanoic acid, 2-hexyl-, methyl ester	113415	010152-61-1	46
20	19.697	0.11	C:\Database\NIST05a.L			
			9-Octadecene, 1,1-dimethoxy-, (Z)-	132340	015677-71-1	7
			1,5-Dichloro-2,3-dinitrobenzene	82706	028689-08-9	4
			2-(2-Thienyl)ethylamine	11425	030433-91-1	4

Library Search Report

Data Path : D:\Data\psm2011\waste cooking oil 4jan12\
 Data File : CD+KOH(2)-biodiesel.D
 Acq On : 4 Jan 2012 10:56
 Operator : FIZA4JAN
 Sample : CD+KOH(2)-biodiesel
 Misc :
 ALS Vial : 5 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
 Integration Events: ChemStation Integrator - autoint1.e

Pk#	RT	Area%	Library/ID	Ref#	CAS#	Qual
21	19.847	0.51	C:\Database\NIST05a.L			
			Eicosanoic acid, methyl ester	140312	001120-28-1	99
			Eicosanoic acid, methyl ester	140314	001120-28-1	99
			Eicosanoic acid, methyl ester	140313	001120-28-1	98
22	19.900	0.18	C:\Database\NIST05a.L			
			9-Octadecenamide, (Z)-	112656	000301-02-0	93
			9-Octadecenamide, (Z)-	112657	000301-02-0	93
			Tetradecanamide	76595	000638-58-4	46
23	20.061	0.06	C:\Database\NIST05a.L			
			Ethanol, 2-bromo-	9919	000540-51-2	23
			3,8-Dioxatricyclo[5.1.0.0(2,4)]octane, 4-ethenyl-	16818	053966-43-1	16
			9-Octadecene, 1,1-dimethoxy-, (Z)-	132340	015677-71-1	12
24	20.221	0.02	C:\Database\NIST05a.L			
			Ethanol, 2-bromo-	9919	000540-51-2	23
			1-Undecanamine	37090	007307-55-3	9
			2,4(1H,3H)-Pyrimidinedione, 6-chloro-5-nitro-	50640	006630-30-4	9
25	20.306	0.08	C:\Database\NIST05a.L			
			Urea, methyl-	755	000598-50-5	9
			N-(2-Thioethyl) N'-methyl urea	14483	072545-68-7	9
			N-(2-Chloroethyl)-N'-methylurea	15455	074378-14-6	9
26	20.701	0.19	C:\Database\NIST05a.L			
			4,7,10,13,16,19-Docosahexaenoic acid, methyl ester, (all-Z)-	148979	002566-90-7	95
			5,8,11,14,17-Eicosapentaenoic acid, methyl ester, (all-Z)-	134757	002734-47-6	93
			1,7-Octadiene, 3,6-dimethylene-	14407	003382-59-0	49
27	20.798	0.18	C:\Database\NIST05a.L			
			1,3-Cyclododecadiene, (E,Z)-	32190	001129-92-6	87
			9,12,15-Octadecatrienoic acid, methyl ester, (Z,Z,Z)-	119876	000301-00-8	59
			5,8,11-Heptadecatrien-1-ol	92210	022117-09-5	55
28	20.990	0.04	C:\Database\NIST05a.L			
			Ethanol, 2-bromo-	9919	000540-51-2	23
			3,8-Dioxatricyclo[5.1.0.0(2,4)]octane, 4-ethenyl-	16818	053966-43-1	9
			(-)-cis-Myrtanylamine	25108	038235-68-6	9
29	21.150	0.13	C:\Database\NIST05a.L			
			No matches found			
30	21.268	0.03	C:\Database\NIST05a.L			
			N-(5-Aminopentyl)-oxalamic acid	39217	1000185-16-0	2

4. Biodiesel from CKD/CH₃OH

Library Search Report

Data Path : D:\Data\psm2011\waste cooking oil 4jan12\
 Data File : CD+CH3OH-biodiesel.D
 Acq On : 4 Jan 2012 11:24
 Operator : FIZA4JAN
 Sample : CD+CH3OH-biodiesel
 Misc :
 ALS Vial : 6 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
 Integration Events: ChemStation Integrator - autoint1.e

Pk#	RT	Area%	Library/ID	Ref#	CAS#	Qual
1	5.373	0.05	C:\Database\NIST05a.L Glycinamide	754	001668-10-6	4
			N-(2-Thioethyl) N'-methyl urea	14483	072545-68-7	3
			Propane	77	000074-98-6	3
2	9.250	0.01	C:\Database\NIST05a.L Propanal, 2,2-dimethyl-, oxime	4023	000637-91-2	9
			Hex-5-enylamine	3452	034825-70-2	4
			3,3-Dimethylbutylamine	4062	015673-00-4	4
3	10.607	0.47	C:\Database\NIST05a.L Dodecanoic acid, methyl ester	67167	000111-82-0	95
			Dodecanoic acid, methyl ester	67169	000111-82-0	93
			Nonanoic acid, methyl ester	37541	001731-84-6	72
4	12.701	0.02	C:\Database\NIST05a.L 1-Hexanamine, N-nitro-	21046	098275-81-1	9
			Ethanol, 2-bromo-	9919	000540-51-2	7
			1,7-Diaminoheptane	13167	000646-19-5	7
5	12.914	1.10	C:\Database\NIST05a.L Methyl tetradecanoate	86753	000124-10-7	95
			Methyl tetradecanoate	86750	000124-10-7	95
			Dodecanoic acid, methyl ester	67169	000111-82-0	53
6	13.929	0.04	C:\Database\NIST05a.L Pentadecanoic acid, methyl ester	96271	007132-64-1	97
			Pentadecanoic acid, methyl ester	96270	007132-64-1	95
			Methyl 9-methyltetradecanoate	96259	213617-69-7	91
7	14.826	3.19	C:\Database\NIST05a.L 7-Hexadecenoic acid, methyl ester, (Z)-	104151	056875-67-3	99
			9-Octadecenoic acid (Z)-, methyl ester	122321	000112-62-9	99
			9-Hexadecenoic acid, methyl ester, (Z)-	104152	001120-25-8	99
8	15.553	30.03	C:\Database\NIST05a.L Hexadecanoic acid, methyl ester	105639	000112-39-0	87
			Pentadecanoic acid, 14-methyl-, methyl ester	105659	005129-60-2	70
			Hexadecanoic acid, 2-methyl-	105633	027147-71-3	58
9	16.183	0.11	C:\Database\NIST05a.L 1-Azacyclononan-2-one	18380	000935-30-8	9
			9-Octadecene, 1,1-dimethoxy-, (Z)-	132340	015677-71-1	7
			Cyclopentanol, 2-(aminomethyl)-, trans-	7660	040482-02-8	5
10	16.578	0.12	C:\Database\NIST05a.L Heptadecanoic acid, methyl ester	114853	001731-92-6	95
			Hexadecanoic acid, 15-methyl-, methyl ester	114865	006929-04-0	95
			Heptadecanoic acid, methyl ester	114852	001731-92-6	94
11	17.315	0.13	C:\Database\NIST05a.L 4,9-Decadienoic acid, 2-nitro-, methyl ester	85828	029085-46-9	43

Library Search Report

Data Path : D:\Data\psm2011\waste cooking oil 4jan12\
 Data File : CD+CH3OH-biodiesel.D
 Acq On : 4 Jan 2012 11:24
 Operator : FIZA4JAN
 Sample : CD+CH3OH-biodiesel
 Misc :
 ALS Vial : 6 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
 Integration Events: ChemStation Integrator - autoint1.e

PK#	RT	Area%	Library/ID	Ref#	CAS#	Qual
			Phenylethanolamine	16140	007568-93-6	14
			1,4-Cyclohexanedimethanamine	19170	002549-93-1	9
12	17.646	1.51	C:\Database\NIST05a.L			
			11,14-Octadecadienoic acid, methyl ester	121099	056554-61-1	99
			9,12-Octadecadienoic acid (Z,Z)-, methyl ester	121107	000112-63-0	99
			9,12-Octadecadienoic acid, methyl ester	121093	002462-85-3	99
13	18.309	53.09	C:\Database\NIST05a.L			
			9-Octadecenoic acid, methyl ester, (E)-	122326	001937-62-8	99
			8-Octadecenoic acid, methyl ester	122297	002345-29-1	99
			9-Octadecenoic acid, methyl ester	122299	002462-84-2	99
14	18.437	5.18	C:\Database\NIST05a.L			
			Octadecanoic acid, methyl ester	123709	000112-61-8	94
			Heptadecanoic acid, 14-methyl-, methyl ester	123731	002490-23-5	93
			Heptadecanoic acid, 14-methyl-, methyl ester, (+/-)-	123733	057274-45-0	93
15	18.554	0.06	C:\Database\NIST05a.L			
			1-Heptadecanamine	95504	004200-95-7	30
			Tridecylamine	56578	002869-34-3	22
			1,8-Diaminooctane	20253	000373-44-4	17
16	18.586	0.08	C:\Database\NIST05a.L			
			Ethanol, 2-bromo-	9919	000540-51-2	17
			Octopamine	25339	000104-14-3	10
			Ethanol, 2-bromo-	9920	000540-51-2	9
17	18.715	0.10	C:\Database\NIST05a.L			
			1,4-Cyclohexanedimethanamine	19170	002549-93-1	10
			Ethanol, 2-bromo-	9920	000540-51-2	9
			1,7-Diaminoheptane	13167	000646-19-5	9
18	18.853	0.08	C:\Database\NIST05a.L			
			1-Tetradecanamine	66606	002016-42-4	12
			1,7-Diaminoheptane	13167	000646-19-5	9
			Ethanol, 2-bromo-	9919	000540-51-2	9
19	19.099	0.08	C:\Database\NIST05a.L			
			Adenosine, 2-methyl-	112205	016526-56-0	9
			Urea, octadecyl-	132227	002158-08-9	9
			Urea, butyl-	7773	000592-31-4	9
20	19.206	0.03	C:\Database\NIST05a.L			
			9-Octadecene, 1,1-dimethoxy-, (Z)-	132340	015677-71-1	27
			Ethanol, 2-bromo-	9919	000540-51-2	25
			Ethanol, 2-bromo-	9920	000540-51-2	9
21	19.281	0.57	C:\Database\NIST05a.L			
			5,8,11,14-Eicosatetraenoic acid, ethyl ester, (all-Z)-	143836	001808-26-0	96
			1,3-Cyclododecadiene, (E,Z)-	32190	001129-92-6	74
			Ethyl 5,8,11,14,17-icosapentaenoate	142611	084494-70-2	72

Library Search Report

Data Path : D:\Data\psm2011\waste cooking oil 4jan12\
 Data File : CD+CH3OH-biodiesel.D
 Acq On : 4 Jan 2012 11:24
 Operator : FIZA4JAN
 Sample : CD+CH3OH-biodiesel
 Misc :
 ALS Vial : 6 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
 Integration Events: ChemStation Integrator - autoint1.e

PK#	RT	Area%	Library/ID	Ref#	CAS#	Qual
22	19.345	0.13	C:\Database\NIST05a.L 4,9-Decadienoic acid, 2-nitro-, et hyl ester	85828	029085-46-9	25
			4,9-Decadienoic acid, 2-nitro-, et hyl ester	85830	029085-46-9	23
			Benzeneethanamine, .beta.-methyl-	15120	000582-22-9	16
23	19.430	0.61	C:\Database\NIST05a.L 9,12,15-Octadecatrienoic acid, met hyl ester, (Z,Z,Z)-	119876	000301-00-8	93
			7,10,13-Eicosatrienoic acid, methy l ester	137079	030223-51-9	93
			Sulfuric acid, 5,8,11-heptadecatri enyl methyl ester	141217	056554-67-7	72
24	19.569	0.33	C:\Database\NIST05a.L 11,13-Eicosadienoic acid, methyl e ster	138090	056599-57-6	99
			11,14-Eicosadienoic acid, methyl e ster	138089	002463-02-7	97
			10,13-Eicosadienoic acid, methyl e ster	138091	030223-50-8	96
25	19.633	0.44	C:\Database\NIST05a.L 11-Eicosenoic acid, methyl ester	139171	003946-08-5	99
			Cyclododecanemethanol	55891	001892-12-2	35
			2-Cyclopenten-1-one, 2-pentyl-	24112	025564-22-1	30
26	19.719	0.23	C:\Database\NIST05a.L 9-Octadecenoic acid (Z)-, methyl e ster	122323	000112-62-9	99
			9-Octadecenoic acid (Z)-, methyl e ster	122321	000112-62-9	99
			9-Octadecenoic acid, methyl ester, (E)-	122326	001937-62-8	99
27	19.847	0.32	C:\Database\NIST05a.L Eicosanoic acid, methyl ester	140314	001120-28-1	98
			Eicosanoic acid, methyl ester	140312	001120-28-1	98
			Eicosanoic acid, methyl ester	140313	001120-28-1	98
28	19.943	0.71	C:\Database\NIST05a.L 9-Octadecenamide, (Z)-	112657	000301-02-0	99
			9-Octadecenamide, (Z)-	112655	000301-02-0	93
			9-Octadecenamide, (Z)-	112656	000301-02-0	64
29	20.093	0.16	C:\Database\NIST05a.L 3,8-Dioxatricyclo[5.1.0.0(2,4)]oct ane, 4-ethenyl-	16818	053966-43-1	10
			Ethanol, 2-bromo-	9919	000540-51-2	9
			2,4,6-Octatrien-1-ol, 3,7-dimethyl -(E,E)-	24183	089155-85-1	7
30	20.306	0.17	C:\Database\NIST05a.L Heptanedioic acid, dimethyl ester	49052	001732-08-7	9
			Propanamide, N-(2-hydroxyethyl)-	8259	018266-55-2	5
			Propanoic acid	794	000079-09-4	4

Library Search Report

Data Path : D:\Data\psm2011\waste cooking oil 4jan12\
 Data File : CD+CH3OH-biodiesel.D
 Acq On : 4 Jan 2012 11:24
 Operator : FIZA4JAN
 Sample : CD+CH3OH-biodiesel
 Misc :
 ALS Vial : 6 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
 Integration Events: ChemStation Integrator - autoint1.e

PK#	RT	Area%	Library/ID	Ref#	CAS#	Qual
31	20.573	0.04	C:\Database\NIST05a.L 9-Octadecene, 1,1-dimethoxy-, (Z)- Cyclododecanol, 1-aminomethyl- Ethanol, 2-bromo-	132340 66564 9919	015677-71-1 000832-29-1 000540-51-2	25 17 12
32	20.734	0.23	C:\Database\NIST05a.L 4,7,10,13,16,19-Docosahexaenoic ac id, methyl ester, (all-Z)- 5,8,11,14,17-Eicosapentaenoic acid , methyl ester, (all-Z)- 1,7-Octadiene, 3,6-dimethylene-	148979 134757 14407	002566-90-7 002734-47-6 003382-59-0	93 87 49
33	20.830	0.08	C:\Database\NIST05a.L 4,9-Decadienoic acid, 2-nitro-, et hyl ester 3,8-Dioxatricyclo[5.1.0.0(2,4)]oct ane, 4-ethenyl- 2,4,6-Octatrien-1-ol, 3,7-dimethyl -(E,E)-	85828 16818 24183	029085-46-9 053966-43-1 089155-85-1	17 10 10
34	20.862	0.09	C:\Database\NIST05a.L 4,9-Decadienoic acid, 2-nitro-, et hyl ester 3,8-Dioxatricyclo[5.1.0.0(2,4)]oct ane, 4-ethenyl- Bicyclo[3.1.1]hept-3-ene-spiro-2,4 '-(1',3'-dioxane), 7,7-dimethyl-	85828 16818 52732	029085-46-9 053966-43-1 1000149-76-2	36 12 12
35	21.022	0.03	C:\Database\NIST05a.L Ethanol, 2-bromo- Cyclododecanol, 1-aminomethyl- 1,4-Cyclohexanedimethanamine	9919 66564 19170	000540-51-2 000832-29-1 002549-93-1	33 10 9
36	21.225	0.40	C:\Database\NIST05a.L No matches found			

5. Biodiesel production from CKD/H₂O

Library Search Report

Data Path : D:\Data\psm2011\waste cooking oil 4jan12\
 Data File : CD+H2O(2)-biodeiesel.D
 Acq On : 4 Jan 2012 12:46
 Operator : FIZA4JAN
 Sample : CD+H2O(2)-biodeiesel
 Misc :
 ALS Vial : 9 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
 Integration Events: ChemStation Integrator - autoint1.e

Pk#	RT	Area%	Library/ID	Ref#	CAS#	Qual
1	10.607	0.37	C:\Database\NIST05a.L Dodecanoic acid, methyl ester Dodecanoic acid, methyl ester Dodecanoic acid, methyl ester	67167 67169 67168	000111-82-0 000111-82-0 000111-82-0	94 93 72
2	12.701	0.01	C:\Database\NIST05a.L 1,7-Diaminoheptane Ethanol, 2-bromo- 2-(3-Methylguanidino)ethanol	13167 9919 8202	000646-19-5 000540-51-2 1000242-22-5	7 5 5
3	12.914	1.01	C:\Database\NIST05a.L Methyl tetradecanoate Methyl tetradecanoate Methyl tetradecanoate	86753 86750 86752	000124-10-7 000124-10-7 000124-10-7	95 95 95
4	13.929	0.03	C:\Database\NIST05a.L Heptanedioic acid, dimethyl ester 1-Pentanamine Urea, methyl-	49052 1874 756	001732-08-7 000110-58-7 000598-50-5	16 9 9
5	14.816	0.60	C:\Database\NIST05a.L 7-Hexadecenoic acid, methyl ester, (Z)- 9-Hexadecenoic acid, methyl ester, (Z)- 11-Hexadecenoic acid, methyl ester	104151 104152 104135	056875-67-3 001120-25-8 055000-42-5	99 99 93
6	14.869	0.60	C:\Database\NIST05a.L 7-Hexadecenoic acid, methyl ester, (Z)- 9-Hexadecenoic acid, methyl ester, (Z)- 11-Hexadecenoic acid, methyl ester	104151 104152 104135	056875-67-3 001120-25-8 055000-42-5	99 99 93
7	14.944	0.67	C:\Database\NIST05a.L 7-Hexadecenoic acid, methyl ester, (Z)- 9-Hexadecenoic acid, methyl ester, (Z)- 11-Hexadecenoic acid, methyl ester	104151 104152 104135	056875-67-3 001120-25-8 055000-42-5	99 99 94
8	15.585	31.68	C:\Database\NIST05a.L Hexadecanoic acid, methyl ester Pentadecanoic acid, 14-methyl-, methyl ester Pentadecanoic acid, 13-methyl-, methyl ester	105639 105659 105660	000112-39-0 005129-60-2 005487-50-3	87 64 49
9	16.226	0.06	C:\Database\NIST05a.L Tridecylamine Cyclopentanemethanamine, 2-amino- Ethanol, 2-bromo-	56579 7186 9919	002869-34-3 021544-02-5 000540-51-2	9 9 7
10	16.664	0.09	C:\Database\NIST05a.L Acetamide, N-ethyl- Acetamide, N-ethyl- Acetamide, N-ethyl-	1850 1846 1849	000625-50-3 000625-50-3 000625-50-3	9 9 7
11	18.351	55.13	C:\Database\NIST05a.L			

Library Search Report

Data Path : D:\Data\psm2011\waste cooking oil 4jan12\
 Data File : CD+H20(2)-biodeiesel.D
 Acq On : 4 Jan 2012 12:46
 Operator : FIZA4JAN
 Sample : CD+H20(2)-biodeiesel
 Misc :
 ALS Vial : 9 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
 Integration Events: ChemStation Integrator - autoint1.e

PK#	RT	Area%	Library/ID	Ref#	CAS#	Qual
			9-Octadecenoic acid, methyl ester, (E)-	122326	001937-62-8	99
			8-Octadecenoic acid, methyl ester	122297	002345-29-1	99
			10-Octadecenoic acid, methyl ester, (E)-	122330	013038-45-4	99
12	18.469	5.36	C:\Database\NIST05a.L Octadecanoic acid, methyl ester	123709	000112-61-8	94
			Heptadecanoic acid, 14-methyl-, methyl ester, (+/-)-	123733	057274-45-0	93
			Heptadecanoic acid, 14-methyl-, methyl ester	123731	002490-23-5	93
13	18.725	0.08	C:\Database\NIST05a.L 1,4-Cyclohexanedimethanamine	19170	002549-93-1	35
			3,8-Dioxatricyclo[5.1.0.0(2,4)]octane, 4-ethenyl-	16818	053966-43-1	12
			Cyclohexanol, 2-(aminomethyl)-, trans-	12596	005691-09-8	9
14	18.864	0.06	C:\Database\NIST05a.L Ethanol, 2-bromo-	9919	000540-51-2	23
			9-Octadecene, 1,1-dimethoxy-, (Z)-	132340	015677-71-1	9
			N-(3-Methylbutyl)acetamide	12551	013434-12-3	7
15	19.099	0.08	C:\Database\NIST05a.L Urea, octadecyl-	132227	002158-08-9	45
			Acetamide, N-ethyl-	1850	000625-50-3	17
			Propanamide, N-(2-hydroxyethyl)-	8259	018266-55-2	9
16	19.281	0.99	C:\Database\NIST05a.L 1,3-Cyclododecadiene, (E,Z)-	32190	001129-92-6	95
			5,8,11,14-Eicosatetraenoic acid, methyl ester, (all-Z)-	143836	001808-26-0	93
			Methyl eicosa-5,8,11,14,17-pentaenoate	134744	001191-65-7	64
17	19.430	0.68	C:\Database\NIST05a.L 7,10,13-Eicosatrienoic acid, methyl ester	137079	030223-51-9	91
			9,12,15-Octadecatrienoic acid, methyl ester, (Z,Z,Z)-	119876	000301-00-8	86
			Sulfuric acid, 5,8,11-heptadecatrienyl methyl ester	141217	056554-67-7	81
18	19.569	0.32	C:\Database\NIST05a.L 11,14-Eicosadienoic acid, methyl ester	138089	002463-02-7	99
			11,13-Eicosadienoic acid, methyl ester	138090	056599-57-6	99
			10,13-Eicosadienoic acid, methyl ester	138091	030223-50-8	96
19	19.633	0.53	C:\Database\NIST05a.L 11-Eicosenoic acid, methyl ester	139171	003946-08-5	99
			12-Octadecenoic acid, methyl ester	122311	056554-46-2	70
			10-Octadecenoic acid, methyl ester	122312	013481-95-3	70
20	19.719	0.19	C:\Database\NIST05a.L			

Library Search Report

Data Path : D:\Data\psm2011\waste cooking oil 4jan12\
 Data File : CD+H20(2)-biodeiesel.D
 Acq On : 4 Jan 2012 12:46
 Operator : FIZA4JAN
 Sample : CD+H20(2)-biodeiesel
 Misc :
 ALS Vial : 9 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
 Integration Events: ChemStation Integrator - autoint1.e

Pk#	RT	Area%	Library/ID	Ref#	CAS#	Qual
			Nona-2,3-dienoic acid, ethyl ester	44341	1000187-19-2	11
			1-Methyl-2-aminomethylimidazole	5933	124312-73-8	9
			9-Octadecene, 1,1-dimethoxy-, (Z)-	132340	015677-71-1	8
21	19.847	0.46	C:\Database\NIST05a.L			
			Eicosanoic acid, methyl ester	140314	001120-28-1	98
			Eicosanoic acid, methyl ester	140313	001120-28-1	98
			Eicosanoic acid, methyl ester	140312	001120-28-1	98
22	20.071	0.07	C:\Database\NIST05a.L			
			3,8-Dioxatricyclo[5.1.0.0(2,4)]octane, 4-ethenyl-	16818	053966-43-1	25
			Phenylethanolamine	16140	007568-93-6	10
			1,4-Cyclohexanedimethanamine	19170	002549-93-1	9
23	20.242	0.02	C:\Database\NIST05a.L			
			Hexafluoro-1,5-pentanediol	66139	000376-90-9	10
			9-Octadecene, 1,1-dimethoxy-, (Z)-	132340	015677-71-1	10
			Ethanol, 2-bromo-	9920	000540-51-2	9
24	20.328	0.04	C:\Database\NIST05a.L			
			Propanamide, N-(2-hydroxyethyl)-	8259	018266-55-2	9
			Acetamide, N-ethyl-	1846	000625-50-3	9
			N-n-Butylpropionamide	12534	002955-67-1	9
25	20.563	0.01	C:\Database\NIST05a.L			
			1-Heptadecanamine	95504	004200-95-7	12
			1-Octanamine	12617	000111-86-4	9
			Tridecylamine	56578	002869-34-3	9
26	20.723	0.20	C:\Database\NIST05a.L			
			4,7,10,13,16,19-Docosahexaenoic acid, methyl ester, (all-Z)-	148979	002566-90-7	95
			5,8,11,14,17-Eicosapentaenoic acid, methyl ester, (all-Z)-	134757	002734-47-6	81
			9,12,15-Octadecatrien-1-ol, (Z,Z,Z)-	101506	000506-44-5	46
27	20.819	0.08	C:\Database\NIST05a.L			
			4,9-Decadienoic acid, 2-nitro-, ethyl ester	85828	029085-46-9	12
			3,8-Dioxatricyclo[5.1.0.0(2,4)]octane, 4-ethenyl-	16818	053966-43-1	12
			2-Propynoic acid, 3-(1-hydroxycyclohexyl)-, methyl ester	44255	1000317-29-4	10
28	20.851	0.07	C:\Database\NIST05a.L			
			3,8-Dioxatricyclo[5.1.0.0(2,4)]octane, 4-ethenyl-	16818	053966-43-1	12
			Benzeneethanamine, .beta.-methyl-	15120	000582-22-9	9
			2-(p-Tolyl)ethylamine	15061	003261-62-9	9
29	21.011	0.02	C:\Database\NIST05a.L			
			Ethanol, 2-bromo-	9919	000540-51-2	27
			9-Octadecene, 1,1-dimethoxy-, (Z)-	132340	015677-71-1	16
			(-)-cis-Myrtanylamine	25108	038235-68-6	10
30	21.193	0.46	C:\Database\NIST05a.L			
			1-Azacyclononan-2-one	18382	000935-30-8	9

Library Search Report

Data Path : D:\Data\psm2011\waste cooking oil 4jan12\
Data File : CD+H20(2)-biodeiesel.D
Acq On : 4 Jan 2012 12:46
Operator : FIZA4JAN
Sample : CD+H20(2)-biodeiesel
Misc :
ALS Vial : 9 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e

PK#	RT	Area%	Library/ID	Ref#	CAS#	Qual
			9-Octadecene, 1,1-dimethoxy-, (Z)-	132340	015677-71-1	9
			Furazan, dimethyl-	2971	004975-21-7	4

APPENDIX D**Pictures**

Figure 9: CKD, CKD/KOH, CKD/CH₃OH, and CKD/H₂O catalyst



Figure10: Biodiesel Production