DESIGN AND CONSTRUCTION OF A PILOT PLANT FOR THE PRODUCTION OF BIODIESEL FROM COTTONSEED OIL

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DEDICATION

This thesis is dedicated to

My humble wife Bilkisu, and

My daughter Fatima (Ummi).

CERTIFICATION

This dissertation entitled "Design and fabrication of a pilot plant for the production of biodiesel from cottonseed oil." by Musa, Idris Atadashi meets the regulations governing the award of the degree of Masters of Science in Chemical Engineering of Ahmadu Bello University, Zaria, and is approved for its contribution to knowledge and literary presentation.

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ABSTRACT

A pilot plant was designed and constructed for the production of twenty (20) litres of biodiesel per batch from cottonseed oil. SUPER PRO design software was used to design the transesterification reactor, mixer and heat exchanger. These were constructed locally, the equipment installed, and the plant commissioned. Results of runs carried out indicated that time of reaction greatly affects the yield of the biodiesel and its quality. The results have shown that reaction time of 1hr for transesterification yielded biodiesel of best physico-chemical properties and most importantly yielded 95.02% of methylesters (biodiesel), signifying most profitable operation. The compositional analysis of the methylesters was carried out using Fourier Transform Infrared Spectroscopy. The physico-chemical properties were compared with E.U. (EN14214) and ASTM (D6751) standard properties of biodiesel and found to fall within the standards. The economic analysis indicated that the value of Discounted Cash Flow Rate of Return (DCFRR) was 31.4%, and the Rate of Return (ROR), was 27.5%. The Pay Back Period of this plant has been calculated to be approximately 4 years indicating that the plant is economically viable.

CHAPTER ONE

INTRODUCTION

1.1 Biodiesel Production

Biodiesel is defined as the mono-alkyl esters of fatty acids derived from vegetable oils or animal fats. In simple terms, biodiesel is the product you get when a vegetable oil or animal fat is chemically reacted with an alcohol to produce a new compound that is known as fatty acid alkyl ester. A catalyst such as sodium or potassium hydroxide is required. Glycerol is produced as a byproduct. Biodiesel is an alternative to Automotive Gas Oil (AGO), other wise known as diesel. Chemically, it comprises of a mixture of alkyl esters of long chain fatty acids. The study of biodiesel fuel has been conducted for over 100 years, but interest lagged because of cheap and plentiful supplies of petroleum fuels. Periodic increase in petroleum prices due to more demand, stringent emission norms, and feared shortages of petroleum fuels due to rapid depletion and net production of carbon dioxide (CO_2) from combustion sources have rekindled interest in renewable biodiesel fuels. Since the sharp oil price increase of 1973, various alternative fuels have been investigated with the goal of replacing conventional petroleum supplies (Kusy, 2006). The initial interest was mainly to address the issue of fuel supply security, but recently more attention has been focused on the use of renewable fuels in order to reduce the net production of carbon dioxide (CO_2) from fossil fuel combustion. The main advantages of using biodiesel are its renewability, better quality exhaust gas emission, biodegradability and it does not contribute to a rise in the level of carbon dioxide in the atmosphere (Abreu et al, 2003).

Renewable resources like vegetable oils, for instance cottonseed oil, when converted to biodiesel, take away more carbon dioxide from the atmosphere during their production than is added to it by later combustion. Therefore, it alleviates the increasing carbon dioxide content of the atmosphere (Kusy, 2006). Many investigations revealed that the use of crude vegetable oil directly as fuel in diesel engine created various problems (Siekmann et al, 1982). These include excessive deposits and poor combustion. The high viscosity of vegetable oil was largely responsible for these problems. Reducing the viscosity of vegetable oil by blending, pyrolysis and emulsification did not solve the problem completely (Kusy, 2006). The alternate way is to make use of vegetable oil derivates, namely monoesters, and it has been established that transesterification is among the best ways to produce esters from vegetable oil (Siekmann et al, 1982). Transesterification is a process involving reaction between triglyceride and alcohol in the presence of a catalyst to produce ester and glycerol. The molecular weight of a typical ester is roughly one third that of typical oil molecule and therefore has a much lower viscosity. To complete the transesterification process stoichiometrically, 3:1 molar ratio of alcohol to triglycerides is needed. However in practice, higher ratio of alcohol to oil ratio is generally employed to ensure completion of the reaction (Moore and Davies, 1994).

Alcohols are primary and secondary monohydric aliphatic compounds having 1-8 carbon atoms. Among the alcohols that can be used in the transesterification process are methanol, ethanol, propanol, butanol, and amyl alcohol. Methanol and ethanol are widely used and especially methanol because of its low cost, and sodium hydroxide (NaOH) easily dissolves in it. Physiochemical properties of methanol include moleculae weight

(32.04), boiling temperatre (64.7^0C) and specific gravity (0.792 gm/ml). As the molecular weight is low compared to other alcohols, less amount of methanol is required on weight basis for the reaction. Since boiling temperature is also low compared to other alcohols, less energy is required for the reaction. Apart from alkali, other catalysts such as acids and enzymes can also be used for transesterification. Some of the alkali based catalysts include NaOH, potassium hydroxide (KOH), carbonates and corresponding sodium methoxide, sodium ethoxide, sodium propoxide and sodium buoxide. Sulphuric acid, sulphinic acids and hydrochloric acid are usually used for acid catalysis. Alkali catalyzed transesterification is much faster than acid catalyzed transesterification and is most often used commercially (Moore and Davies, 1994). Transmethylation occurs approximately 4000 times faster in the presence of alkaline catalyst than those catalyzed by the same amount of acid catalyst (Kusy, 2006). In Biocatalysts, Lipases can be used as catalysts in transesterification reaction. The other important parameter is stirring speed, which plays a vital role in transesterification process (Sankaran, 2006). The degree of homogeneity (emulsification) of alcohol in the triglyceride phase is of great importance in the transesterification reaction (Barnwal and Sharma, 2005). After transesterification, the ester can be separated from glycerol by simple gravitational sedimentation and the ester has to be washed with slightly acidified water to remove traces of alkali (Suppes et al, 2001).

1.2 Environmental and Other Factors

Biodiesel is not only renewable and sustainable energy resource but is nontoxic, unlike the conventional petroleum based diesels. Biodiesel is also biodegradable and its use helps reduce the emission of carbon dioxide into the atmosphere. The Kyoto

agreement (1997), sponsored by the United Nations, has set the limit of carbon dioxide (CO_2) emission into the atmosphere in order to stem the ever-increasing tide of global warming, weather disruptions and the EI Nino and La Nina effects (Howell, 1997). All countries of the world signed the agreement except the USA, whose CO_2 emission on per capita basis is over 20 times that of most developing countries (Fernando and Hanna**,** 2004).

Biodiesel helps in reducing the CO_2 accumulation in the atmosphere because the very process by which plants make their food for growth is photosynthesis, which consumes atmospheric CO_2 in the presence of solar energy (sun shine). Even though CO_2 would be discharged into the atmosphere when the fuels are used in vehicles or for other heating purposes, since some of the plant materials (roots, stems etc.) are still left un-used, the net effect is an overall CO_2 reduction in the atmosphere. The other major advantage of biodiesels is that their use does not lead to emission of sulphur compounds into the atmosphere, unlike petroleum products. Sulphur compounds are not only health hazard to humans, but they also create "acid rain" in some areas of the world.

The development of biodiesel in Nigeria would also lead to job creation in the production, harvesting and processing of these energy materials.

1.3 Research Problem Statement

This research is intended to address the following problems

1 Over reliance on diesel fuel (AGO) has resulted in continuous price hike, and some times scarcity hence becoming imperative to develop alternatives.

2. Fossil fuels are not renewable and are also not environmentally friendly.

1.4 Research Objectives

1. Design and fabricate a biodiesel pilot plant utilising cottonseed oil as feed stock.
2. Operate the pilot plant and collect data for optimization.
3. Characterise the biodiesel produced and compare the results with those of international standards.
4. Economic evaluation of the pilot plant and recommendations.

1.5 Justification

1. A lot of bench scale data has been generated in the past and there is need to take that further in pilot plant studies of biodiesel production in Nigeria.
2. Petroleum availability is finite – Biodiesel from renewable sources ensures energy security for the country and could extend availability to one of the conventional AGO.
3. Generation of employment opportunities, thereby providing livelihood support for areas that produce the feed, cottonseed oil.
4. The Plantations of oil yielding plants such as cottonseed for biodiesel production will result in environmental security

CHAPTER TWO

LITERATURE REVIEW

2.1 Biodiesel Production

Biodiesel is produced by treating oils from seeds (e.g., cottonseed) in transeterification reaction with alcohol. The major products are the monoesters, commonly referred to as "biodiesel." No major engine modifications are necessary to use biodiesel in place of petroleum-based diesel (Ramadhas et al, 2005). Biodiesel can be mixed with petroleum-based diesel in any proportion. Biodiesel is registered as a fuel and fuel additive in US with the Environmental Protection Agency (EPA) and is widely used both in Europe, U.S and some parts of Asia. The use of biodiesel results in a substantial reduction of unburnt hydrocarbons and the overall ozone forming potential of the speciated hydrocarbon emissions from biodiesel is nearly 50 percent less than that measured for conventional diesel fuel (Ramadhas et al, 2005).The flash point of biodiesel has been tested and reported by many sources (De caro et al, 2001). It has been concluded that the flash point ($>130^0$C) of biodiesel blends rises as the percentage of biodiesel increases. Therefore, pure biodiesel or blends of biodiesel with petroleum diesel are safer to store, handle and use than conventional diesel fuel. In addition, it is essentially sulphur-free and eliminates the emission of sulphur dioxide and sulphate aerosols.

Biodiesel can be produced from several oil seeds, such as Cottonseed, Jatropha, Castor seeds, etc. These raw materials are renewable, making biodiesel a renewable fuel and as such a more sustainable energy resource.

2.2 Environmental Considerations

Climate change is presently an important consideration in energy use and sustainable development. Biodiesel is considered environmentally friendly almost all of the carbon dioxide released during consumption had been sequestered out of the atmosphere during crop growth. Combustion of one liter of diesel fuel results in the emission of about 2.6 kilograms of CO_2 (Ramadhas et al, 2005).The amount of CO_2 required to produce the 1 litre is about 5.2 kilograms, therefore, the use of biodiesel will directly reduce the amount of CO_2 in the atmosphere.

Combustion of biodiesel has been reported in a number of sources (Moore and Davies, 1994), to have lower emissions compared with conventional Automotive Gas Oil (AGO). Lower emission of SO_2, soot, carbon monoxide (CO), hydrocarbons (HC), polyaromatic hydrocarbons (PAH), and aromatics are presented in Figure 2.1. Oxides of nitrogen (NO_X) emissions from biodiesel are reported to range between plus or minus 10% that of AGO depending on engine combustion characteristics (Tickell, 1999).

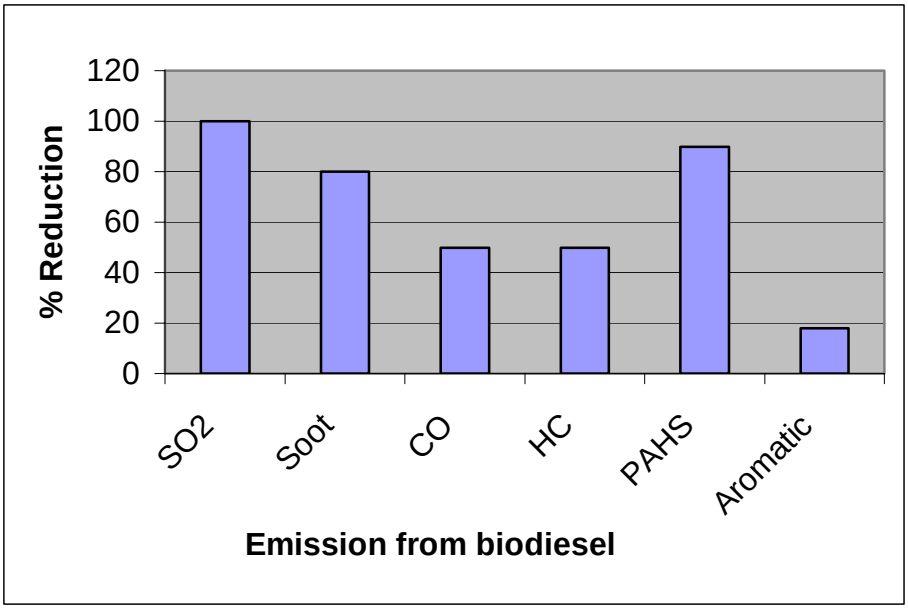


Figure 2. 1: Lower Emissions of Biodiesel compared with Petrodiesel.

Source: (Tickell, 1999).

Biodiesel almost completely eliminates lifecycle carbon dioxide emissions. When compared to petro-diesel it reduces emission of particulate matter by 40%, unburnt hydrocarbons by 68%, carbon monoxide by 44%, sulphates by 100%, polycyclic aromatic hydrocarbons (PAHS) by 80%, and the carcinogenic-nitrated PAHS by 90% on an average (Tickell, 1999).

2.3 Advantages and Disadvantages of Biodiesel

Besides environmental consideration, biodiesel use has other advantages. The higher cetane number of biodiesel compared to petro-diesel indicates potential for higher engine performance. Tests have shown that biodiesel has similar or better fuel consumption, horsepower, and torque and haulage rates as conventional diesel (De Caro et al, 2001). The superior lubricating properties of biodiesel further increases functional engine efficiency. Their higher flash point makes them safer to store. The biodiesel molecules are simple hydrocarbon chains, containing no sulfur,

or aromatic substances associated with fossil fuels. They contain higher amounts oxygen (up to 10%) that ensures more complete combustion of hydrocarbons.

Production of biodiesel leads to the production of many by- products for example, 1 t/ ha/yr of high protein seed cake (60% crude protein) that can be potentially used as animal and fish feeds. Also organic matter that could be used as fertilizer particularly in remote areas is achieved in the production of biodiesel from Cottonseed oil. Various other products from the plant (leaf, bark and seed extracts have other industrial and pharmaceutical uses (Abreu et al, 2003).

Inspite of the many advantages in the use of biodiesel; nevertheless, there are some disadvantages which are being overcome. Some of these include:

- High cost of production: This will eventually solve itself when large-scale production and use starts.
- Modifications are required to the automobiles for use of 100% biodiesl: many automobile brands are currently marketed ready for use with biodiesel (De Caro et al, 2001).
- High CFPP (cold filter plugging point) values and hence solidification and clogging of the system at low temperatures: this problem occurs only in tropical countries where the temperature goes down to sub-zero centigrade. Even there, the problem is currently reduced by use of additives (De Caro et al, 2001).

Table 2.1 shows the European Biodiesel physico-chemical properties standard specification (Fernando and Hanna, 2004).

Table 2.1: EU Standard Properties of Biodiesel and Automotive Gas Oil

PROPERTY	UNITS	EU Standards (EN 14214)	EU Standards for AGO
Density@ 30^{o}C	g/ml	>0.8	0.82-0.865
Combustion point	^{o}C	>55	-
Kinematic viscosity	Mm^2/s	5	1.9-5
Calorific value	kJ/kg	Undefined	7500-8500
Cetane number	-	>55	48
Ester content	%	>99	-
Sulfur content	%	<.055	<0.5
Carbon residue	%	<0.1	<0.3

In general, the standard storage and handling procedures used for petroleum diesel can be used for biodiesel, and should be done in a clean dry, dark environment. Acceptable storage tank materials include aluminum, steel, polyethylene, and polypropylene. Teflon, copper, brass, lead, tin and zinc should be avoided (Fernando and Hanna, 2004)**.**

2.4 Vegetable Oils

Vegetable oils are produced from numerous oil seed crops. While all vegetable oils have high energy content, most require some processing to ensure safe use in internal combustion engines. Some of the sources of vegetable oil include sunflower, cottonseed oil, and soybean oil. Vegetable oils and their esters are found to be promising alternatives as fuel for diesel engines. Because of their high viscosity, drying with time and thickening in cold conditions, direct use of vegetable oil as fuels have problems, such as flow, atomization and heavy particulate emissions. As such it makes better sense to convert them to biodiesel by transesterification (Siekmann, 1982).

2.4.1 Cottonseed Oil

Cottonseed oil is a vegetable oil extracted from the seeds of cotton plant after the cotton lint has been removed. Figure 2.2 shows a typical cotton flower with linter and seeds. The oil is extracted from the seed and is usually refined to remove gossypol, a naturally occurring toxin that protects the cotton plant from insect damage. Therefore, unrefined cottonseed oil is sometimes used as a pesticide (Knothe et al, 1997). In its natural unhydrogenated state, cottonseed oil, like all other vegetable oils, has no cholesterol. It also contains no trans-fatty acids. However, it does contain over 50% Omega-6 fatty acids and only trace amounts of Omega-3 fatty acids, and the imbalance is considered unhealthy if not used in moderation or balanced elsewhere in the diet (Knothe et al, 1997).



Figure 2.2: Cottonseed Fruits containing both Cotton Linter and Seeds.

Cottonseed oil is rich in palmitic acid (22-26%), oleic acid (15-20%), linoleic acid (49-58%) and 10% mixture of arachidic acid, behenic acid and lignoceric acid. It also contains about 1% sterculic acids and malvalic acids in the crude oil. The cyclopropene

acids are undesirable components, but they are largely removed during refining. Typical composition of cottonseed oil is indicated in Table 2.2.

Table 2.2: The Chemical Analysis of Cottonseed oil (Knothe et al, 1997).

Typical Fatty Acid Composition (%)			
Myristic acid (C14:0)	0.5-2.0	Oleic acid (C18:1)	13-44
Palmitic acid (C16:0)	1.7-29	Linoleic acid (C18:2)	40-63
Palmetoleic acid (C16:1)	<1.5	Linolenic acid (C18:3)	0.1-2.1
Stearic acid (C18:0)	1.0-4.0		

Cottonseed oil is commonly used as cooking oil. Along with soybean oil, it is often partially or fully hydrogenated. The growing consensus is that in hydrogenated (trans-fat) form these oils are unhealthy (Abreu et al, 2003).

Cottonseed oil also costs less than many other varieties, such as palm oil, palm kernel etc., making it a hit amongst commercial restaurant owners and snack food manufacturers (Abreu et al, 2003). Nigeria produces 37.65 million litres of cottonseed oils yearly. The seeds are processed by many factories producing oil and cake (used for animal feed) (Abreu et al, 2003).

Cottonseed oil has many applications, both in the home and in commercial food industries. As a salad oil, it is used in mayonnaise, salad dressings, sauces, and marinades. As cooking oil, it is used for frying in both commercial and home cooking. As a shortening or margarine, it is ideal for baked goods and cake icings (Abreu et al, 2003).

Cottonseed oil has a mild, nut like taste. It is generally clear with a light golden color, but like most oils, the degree of color depends on the amount of refining. Clear, colorless oils are not necessarily better oils, but may have been refined more severely [Ma and Hanna, 1999]. It has a 2:1 ratio of polyunsaturated to saturated fatty acids. Its fatty acid profile generally consists of 70% unsaturated fatty acids including 18% monounsaturated (oleic) and 52% polyunsaturated (linoleic) and 26% saturated (primarily palmitic and stearic) (Mittelbach and Trithart, 1998).

2.4.2 Other Vegetable Oils.

Many other vegetable oils can be used for the production of biodiesel. These include *Jatropha caucus, Ricinus communis* (castor oil), Soya beans oil, Palm oil, Jojoba, etc. Many of these oils are available in Nigeria. Some are already in use as cooking oil, such as Palm and Soya bean oils; others such as Jatropha and castor oil are non-edible and grow in the wild without any established industries extracting their oils.

In the choice of which oil to use, consideration has to be given to a number of factors; such as

(i) Cost of the oil (per kg or litre)

(ii) Physical properties and fatty acid composition of the oil.

(iii) Oil yields from seeds

(iv) Seed yields per hectre of the plant species producimg seeds.

In this work, Cottonseed was selected because of its relative abundance. The analysis of Cottonseed shows the following compositions (Suppes et al, 2001).

Table 2.3: Cottonseed Composition

Parameter	**%**
Moisture	6.20
Protein	18.00
Fats	38.0
Carbonhydrates	17.00
Fibre	15.50
Ash	5.30
Oil content (Seed)	25-30
Oil content (kernel)	50-60
Saturated fatty acids	21
Unsaturated fatty acids	79

In comparing oil yields, Table 2.4 gives conservative estimates for various oil crops (Demirbas, 2003).

Table 2.4: Oil Yields and Characteristics (Demirbas, 2003).

Crop	Kg oil/ha	Litres oil/ha
Corn (Maize)	145	172
Cashew nut	148	176
Oats	183	217
Soy bean	375	446
Sunflowers	800	952
Jojoba	1528	1818
Cottonseed	**1590**	**1892**
Coconut	2260	2689
Oil palm	5000	5950

Oil palm gives the highest oil yield per hectare. However, its wide use as cooking oil makes it more expensive than the other next contenders. Cottonseed oil is generally cheaper than palm oil or Coconut oil and has fairly good oil yield per hectare. It is also readily available in Nigeria.

2.5 Transesterification Reaction

The Transesterification reaction is as follows

CH_2**COOR1**

|

CHCOOR1 + $3CH_3$**OH** → $(CH_2$**OH**$)_2$CH-OH + $3CH_3$**COO-R1**- - - - - - -**1**

|

CH_2COOR1

i.e

Triglyceride + methanol → Glycerol + Esters.

The important factors that affect transesterification reaction are: the amount of methanol and sodium hydroxide, reaction temperature, reaction time, and methanol to oil ratio (Moore and Davies, 1994).

During the esterification process, the triglyceride is reacted with alcohol in the presence of a catalyst, usually a strong alkaline (NaOH, KOH or Na_2SiO_3).

The alcohol reacts with the fatty acids to form the mono-alkyl ester (or biodiesel) and crude glycerol. The reaction between the biolipid (fat or oil) and the alcohol is a reversible reaction so the alcohol must be added in excess to drive the reaction forward and ensure complete conversion.

This reaction is catalysed using the strong bases such as NaOH, KOH, CH_3ONa, etc. Commonly the base (KOH, NaOH) is dissolved in the alcohol to make a convenient method of dispersing the otherwise solid catalyst into the oil. The ROH needs to be very dry. Any water in the process promotes the saponification reaction and inhibits the transesterification reaction (Sridharan and Mathai, 2006).

The reaction mixture is kept just below the boiling point of the alcohol (around 60°C) to speed up the reaction though some workers recommended that the reaction can take place anywhere from room temperature to 55 °C for safety reasons (Sridharan and Mathai, 2006).

Recommended reaction time varies from 1 to 8 hours. Under normal conditions the reaction rate will double with every 10 °C increase in reaction temperature (Knothe, 1997). Excess alcohol is normally used to ensure total conversion of the fat or oil to its esters.

After the reaction is completed, the glycerin phase is much denser than biodiesel phase and the two can be separated by gravity with glycerin simply drawn off from the bottom of the settling vessel. In some cases, a centrifuge is used to separate the two materials faster.

Once the glycerin and biodiesel phases have been separated, the excess alcohol in each phase is removed by distillation. In other systems, the alcohol is removed and the mixture neutralized before the glycerin and esters are separated. In either case, the alcohol is recovered and re-used.

The glycerin by-product contains unused catalyst and soaps that are neutralized with an acid and sent to storage as crude glycerin (water and alcohol are removed later, chiefly using evaporation, to produce 80-88% pure glycerin). Once separated from the glycerin, the biodiesel is sometimes purified by washing gently with warm water to remove residual catalyst or soaps. It is then dried, and sent to storage.

2.6 Production Process

Biodiesel is a liquid fuel produced from vegetable oils by replacing glycerol with a short chain alcohol such as methanol or ethanol in a process known as transesterification as described in section 2.5 (De Caro et al, 2001). The major unit processes involved in a pilot scale production are: raw material purification, tranesterification reaction, seperation and drying. These are described briefly as follows:-

2.6.1 Purification of Raw Materials

The oil to be used in the reactor must be purified by removing all foreign materials. If waste vegetable oil is used, it is filtered to remove dirt, charred food, and other non-oil material often found. Water is removed because its presence causes the triglycerides to hydrolyze to give salts of the fatty acids instead of undergoing transesterification to give biodiesel (Bondioli, 2004). This is often accomplished by heating the filtered oil to approximately 120°C. At this temperature dissolved or suspended water will boil off. When the water boils, it spatters (chemists refer to it as "bumping"). To prevent injury, this operation should be done in a sufficiently large container (at most two thirds full) which is closed but not sealed.

2.6.2 Formation of Esters

A sample of the purified oil is titrated against a standard solution of base in order to determine the concentration of free fatty acids (RCOOH) present in the vegetable oil. The calculated quantity of base (usually sodium hydroxide) is added slowly to the alcohol and it is stirred until it dissolves. Sufficient alcohol is added to make up three full equivalents of the triglyceride, and an excess is added to drive the reaction to completion.

The solution of sodium hydroxide in the alcohol is then added to a warm solution of the oil, and the mixture is heated (typically 50 °C) for several hours to allow the transesterification reaction to take place. A condenser may be used to prevent evaporative losses of the alcohol. Care must be taken not to create a closed system which may lead to explosion.

2.6.3 Separation and Drying

Once the reaction is complete, the glycerol settles down. When methanol is used, it is reported that an emulsion often forms. This emulsion can be broken by standing, centrifugation, or the addition of a low boiling (easily removed) nonpolar solvent, decanting, and distilling (Fernando and Hanna, 2004)**.**

The top layer, which consists of biodiesel and alcohol, is decanted. The excess alcohol can be distilled off, or it can be extracted with water. If the latter is used, the biodiesel should be dried by distillation or with a drying agent.

2.7 Types of Reactors.

The choice of a reactor type from among the several process reactor geometries and configurations will depend on a number of factors.

1. Operating Mode: Batch or Continuous
2. Number and types of reaction phases.

The possible types of reaction geometries for each of these considerations are presented in Table 5.

Note: L – Liquid, G – Gas, S – Solid

Table 2.5: Classification of Reactors

Reaction Type	Homogeneous	L, G, S	Batch Reactor
			Tubular Reactor
			Backmix Reactor
	Heterogeneous	L/L, L/G	Batch Reactor
			Tubular Reactor
			Column Reactor
			Backmix Reactor
		S/L, S/G, S/L/G	Fixed-bed Reactor
			Fluidized-bed Reactor

It has been established in literature that the biodiesel production process is best run in a semi batch mode, especially for small scale plants. In addition, the condition of the reaction indicates that the reaction is heterogeneous. Therefore, the choice of the biodiesel reactor is expected to be a semi batch reactor in which the reactor feed is added and the products removed in batches. For such a reactor, transverse mixing and heat transfer are crucial operating considerations. In addition, materials are usually mixed uniformly in the reactor, so changes in behavior involving mass transfer do not normally take place.

In using a semi batch reactor, mass transfer limitation encountered as a result of two distinctive phases involved is eliminated by using a stirrer empowered with motor and a reducing gear.

CHAPTER THREE

MATERIALS AND METHODS

This chapter deals with materials and methods including detailed design and construction of the biodiesel pilot plant. The materials of the construction and the basis for selecting them are herewith discussed in the chapter.

3.1 Introduction to Design

The main software employed for the design was Super PRO 5.0 (EPROM programmer) developed by a European company (Xeltek Europe). Hysis, a product of Hypotech of Canada was employed in some aspects of the design. AutoCAD software developed by Microsoft U.S was used in some of the drawings.

3.1.1 Process Selection

The biodiesel production process is well discussed in literature (Kusy, 2006). Accordingly the process flow diagram selected is shown in Figure 3.3.

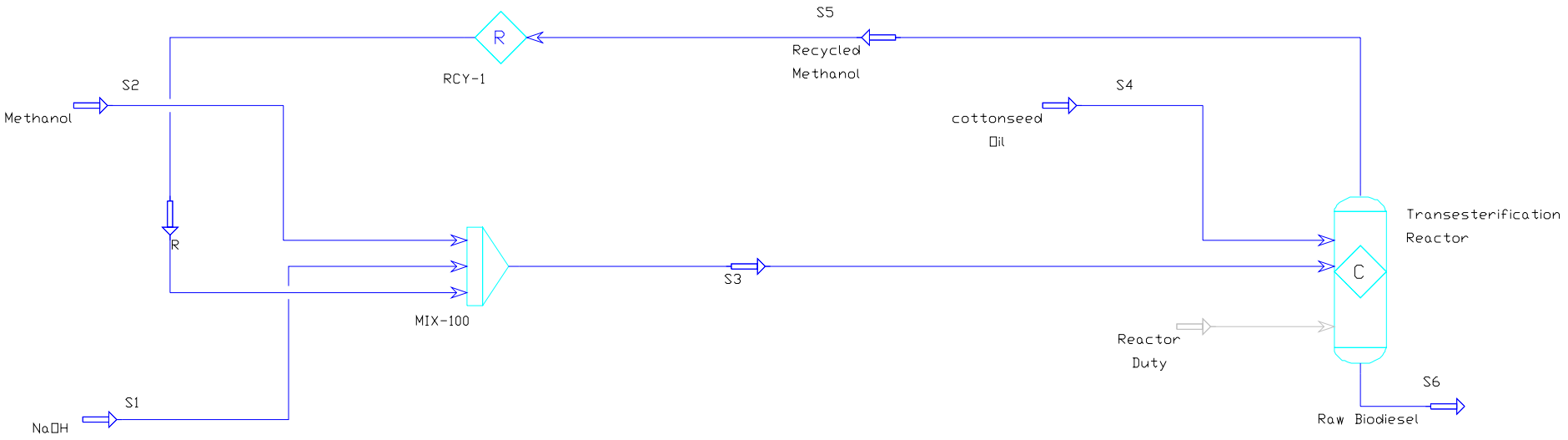


Figure 3.3: Biodiesel Pilot Plant Process Flow Diagram

3.1.2 Material and Energy Balances

To obtain the material and energy balances using Super Pro design software, simulation studies were carried out. The detailed data generated from the simulation is shown in Appendix I.

From this data, the material and energy balances for the pilot plant was obtained and are shown in Table 3.6

Table 3.6: Data for Simulation

Stream No	Material	Temp ^{O}C	Flow (kg/s)	Enthalpies (kJ/kg)
S_1	(Sodium hydroxide)	25	0.1805	
S_2	(Methanol)	25	3.986	(2929)
S_3	Reactor feed	25	4.166	-7239
S_4	Cottonseed oil	25	18.05	-1912
S_5	Recycled methanol	60	0.1030	6231
S_6	Products	60	22.165	2.044

3.2 Reactor Design

The design of the reactor was carried out considering the kinetics of the reaction and the effects of mass and heat transfer. The design involves the determination of the reactor geometry and its sizing, as well as the choice of construction material and the mechanical design of the reactor. The reactor selected is a semi-batch reactor.

3.2.1 Reaction kinetics.

The model used for the correlation of experimental data for the transsterification was based on the proposed thermal transesterification in three reversible steps (Moore and Davies, 1994).

$$TG + CH_3OH \underset{k_2}{\overset{k_1}{\leftrightarrow}} DG + R_1COOCH_3$$

$$DG + CH_3OH \underset{k_4}{\overset{k_3}{\leftrightarrow}} MG + R_2COOH_3$$

$$MG + CH_3OH \underset{k_6}{\overset{k_5}{\leftrightarrow}} GL + R_3COOH_3$$

The following rate equation was used in the design of the transesterification reactor:

$$\frac{d[E]}{dt} = k_1[TG][A] - k_2[DG][E] + k_3[DG][A] - k_4[MG][E] + k_5[MG][A] - k_6[GL][E]$$

Where: TG= Triglyceride, DG= Diglyceride, MG= Monoglyceride, GL= Glycerol,

E= Esters, A= Methanol, and k= Reaction rate constant.

k = Reaction rate constant (available in the library of the software), samples which are indicated in table 3.7.

Table 3.7: Values of Rate Constants.

Rate constants (k)	Values
K_1	1.2734
K_2	2.8632
$K_{3`}$	3.0795
$K_{4`}$	3.9764
K_5	3.9975
K_6	4.0087

3.2.2 Effects of Mass Transfer

Mass transfer problems were eliminated by using a stirrer, thereby decreasing the diffusion resistance to the barest minimum. Reaction rates in these conditions are expected to be determined by chemical kinetic data and not by mass transfer considerations (Ma and Hanna 1999).

3.2.3 Effects of Heat Transfer

The transesterification reaction is reversible and endothermic involving multiple reactions. In addition to this, the endothermic nature of the reaction means that a large amount of heat will be required (see section 3.1) for the desired reaction conditions to be maintained through out the reactor. Steam (low pressure) was used as heating medium to maintain the required reaction temperature.

3.2.4 Design Basis

The reactor for the transesterification process is designed subject to the following:

1. It processes sufficient raw material for the process to produce twenty litres of biodiesel per batch.
2. It has to operate isothermally at a temperature of 60˚C and a pressure of 1bar.
3. The conversion of at least 98% of triglyceride to methyl esters is to be obtained.

3.2.5 Design Parameters

The design parameters are the physical and thermodynamic data of the reactants, and the chemical kinetics of the reaction. The key parameters used in the design are shown in Table 3.8.

Table 3.8: Design Parameters

Physical properties	Cottonseed oil	Methanol
Density 'ρ' (kg/m^3)	915.5	933.1
Viscosity 'μ' (cP)	7375	4.546
Specific heat capacity 'c_p'	1.946	976.4

3.2.6 Determination of Reactor Specifications.

Using Super Pro Design software, the reactor was designed. The parameters shown in Table 3.8 were used and the reactor specifications were determined. Figure 3.4 shows these specifications as determined.

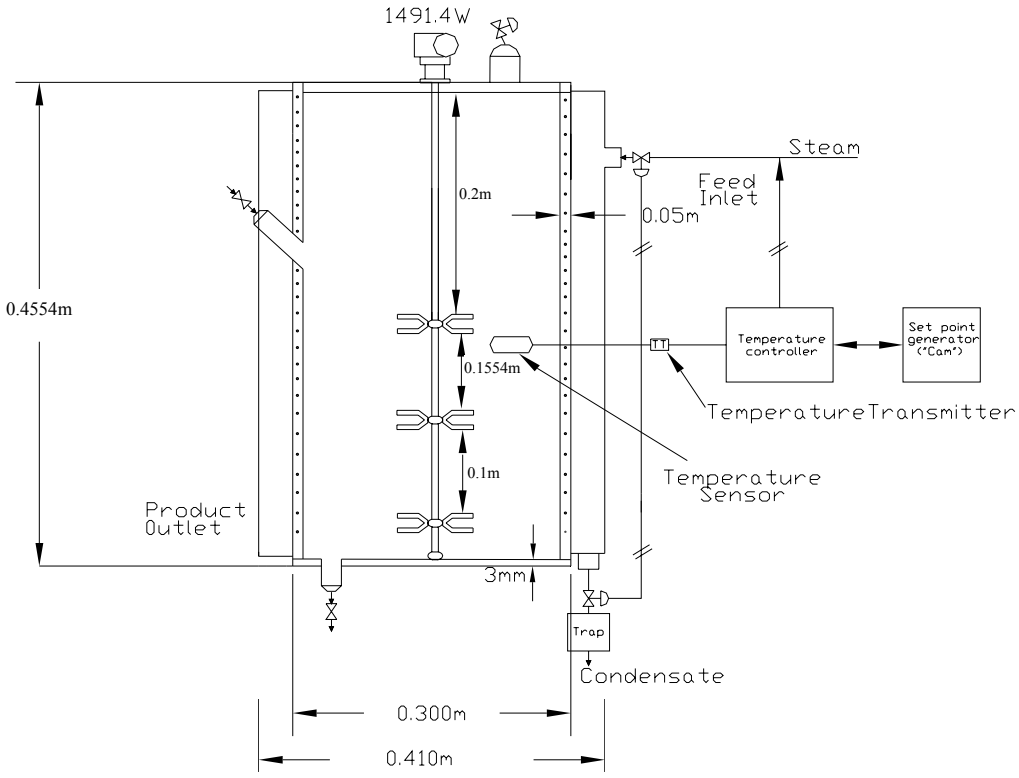


Figure 3.4: Working Drawing of Transesterification Reactor.

3.3 Condenser Design

In the design of the condenser, Hysis software was employed in the design of the condenser. A shell and tube heat exchanger was selected. The design parameters were obtained from the material and energy balances. Likewise, AutoCAD design software

was used for the development of its working drawing. Detailed design specification of the condenser used is shown in Appendix II and the working drawings are shown Figure 3.5.

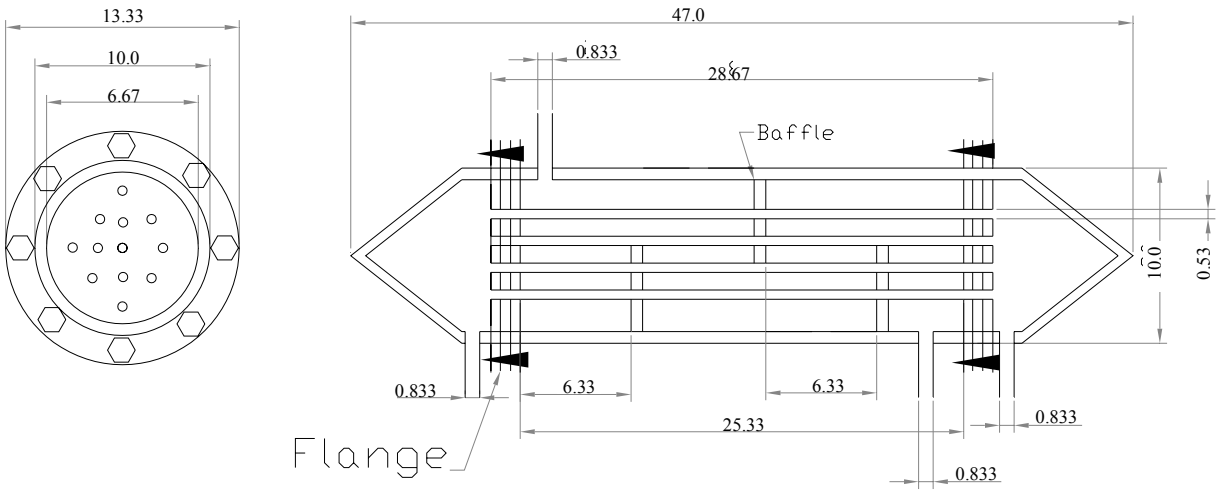


Figure 3. 5: Condenser Working Drawing.

3.3.1 Baffles for the Condenser

Baffles are used to direct fluid to increase shell-side fluid velocity and improve transfer rate. The most common baffle type is the segmental baffle.

Baffle cuts are usually 15-45% of shell diameter, with 20-25% giving optimum results. A baffle cut of 20% is used for the reactor. Baffle Spacing for the reactor is taken as 40% of the shell diameter as this is expected to give optimum results (Ma and Hanna, 1999).

3.4 Mixer Design

The mixer design was carried out using the Super Pro design software. Its working drawings were made possible using AutoCAD design software. Detailed design

specifications of the mixer used is shown in Appendix II and its workings are shown in Figure 3.6.

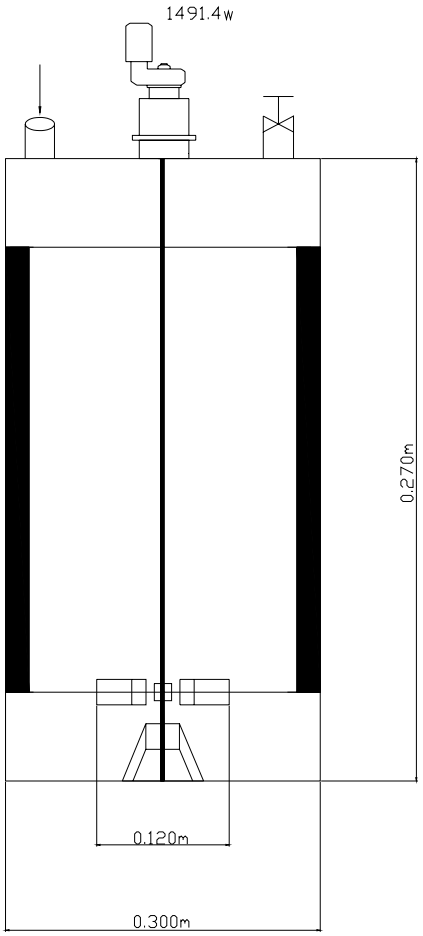


Figure 3.6: Working Drawing of Mixer

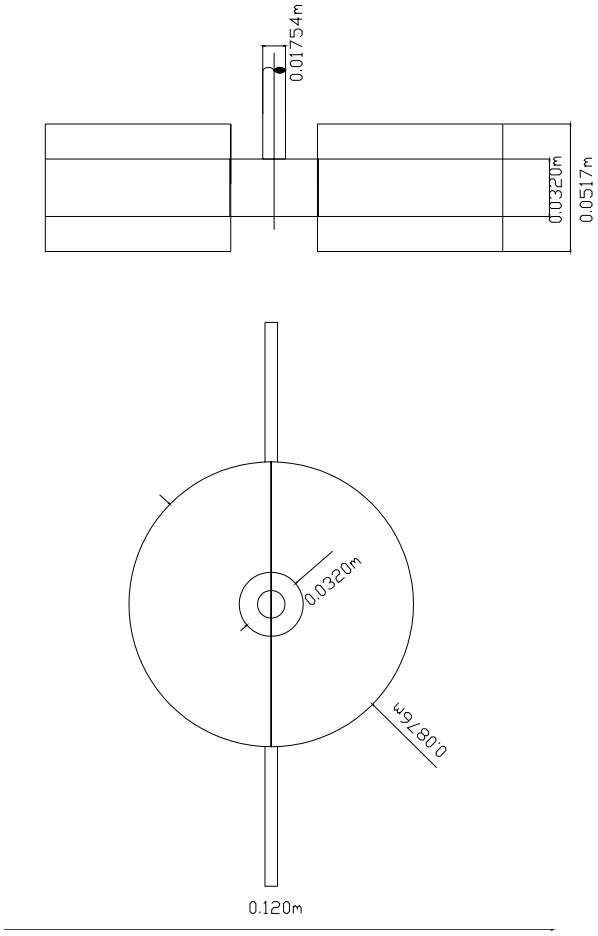


Figure 3.7: Working Drawing of MixersStirrer

3.5 Mechanical Designs

3.5.1 Selection of Motor and Reducing gear

To reduce cost, one motor was used to operate both the reactor and mixer. The selection of the motor and reducing gear and its power rating was done in the design of the two equipment (Reactor and mixer). The power rating was 2 horse powers (1.5Kw) with a reducer of 1:20. Further speed reduction for the mixer was provided by a 1:2 pulley. The motor and the reducing gear is shown in Figure 3.8.



Figure 3.8: Motor and Reducing Gear

3.5.2 Materials of Construction

The material for the construction of the transesterification reactor, mixer, and condenser was design based on a number of factors, most important of which are the design temperature and pressure**,** and the nature of material**s** being handled. Stainless steel was selected for the construction of the pilot plant.

3.5.3 Design Pressure:

This is the maximum pressure to which the reactor and other equipment will be subjected to during operation. The transesterification reactor operates at atmospheric pressure. The design pressure for the transesterification reactor is taken as 1 bar (absolute). However, to avoid pressure build up due to methanol vapourisation, a pressure relief valve was designed to be placed on the reactor at 2 bar (absolute) pressure.

3.5.4 Design Temperature:

Since the strength of metals decreases with increasing temperature, the design temperature is the maximum working temperature that is used to determine the material of construction by its corresponding maximum allowable design stress. The design temperature is chosen as 60˚C for the reactor. This temperature is low enough as not to pose any problem on the operation of the pilot plant.

3.5.5 Corrosion Allowance:

This is additional thickness of metal added to allow for material lost by corrosion and erosion. For low corrosion possibility, a minimum allowance of 2 mm is used. 4 mm is used for more severe corrosion cases (Komers, 2006). The corrosion possibility for this process cannot be adequately predicted from available data and so a corrosion allowance of 3 mm was used. Table 3.9 gives various wall thicknesses for different vessel heights.

Table 3.9: Maximum Practical Wall Thickness [Komers, 2006].

Vessel Height/Length (m)	Wall Thickness (mm)
1.0	5
1.0 to 2.0	7
2.0 to 2.5	9
2.5 to 3.0	10
3.0 to 3.5	12

3.5.6 Design Loads:

The major sources of vessel loads are:

Design Pressure + Liquid Static Head (if significant)

Maximum weight of vessel + Content, under operating conditions

Maximum weight of vessel + Content, under hydraulic test conditions

Wind Loads

Earthquake (seismic loads)

Loads supported by, or resting on the vessel.

These loads were not taken into consideration in the design of the reactor.

3.5.7 Heads/Closures

There are different shapes for cylindrical vessels,

- Flat heads
- Hemispherical heads
- Ellipsoidal heads
- Torispherical heads

The flat head is used for low-pressure applications.

3.5.8 Supports

The kind of support on which the reactor vessel in placed depends on a number of factors. Those factors to be considered here are:

- Size, weight and shape;
- Design temperature, and pressure;

- Internal and external fittings and attachments.

A vertical arrangement was chosen for the reactor vessels due to pressure drop considerations. This required any of two types of supports:

- Saddle supports;
- Brackets/lugs.

The vessel for the reactor is determined to be of moderate size. Thus, saddle support was selected.

3.6 Design Summary

The design of the transesterification reactor, Condenser, and Mixer for the biodiesel process has been carried out. The design specifications are shown in Tables 9-11.

Table 3.10: Transesterification Reactor Design Specifications

Parameter	**Dimensions**
Height of the vessel (m)	0.4554
External diameter (m)	0.300
Internal diameter (m)	0.297
Vessel volume (m^3)	0.03296
Reactor design temperature (^oC)	60
Reactor design pressure (bar)	1
Head thickness (m)	0.005
Shell thickness (m)	0.003
Reactor shell material	Stainless steel (304)

Table 3.11: Condenser Design Specifications

Parameter	Shell side	Tube side
Length (mm).	470	335
Pitch (mm).		7.934
Velocity (kg/hr).	7.20	0.1030
Average Design temp. (^{o}C).	20	60
Design pres. (bar).	1	1
Number of passes per shell.	1	8
Corrosion allowance	1.59	1.59
Pressure drop	0.1	0.1
Material used	Stainless steel (304)	Stainless steel (3040

Table 3.12: Mixer Design Specifications

Parameter	Dimensions
Internal diameter (m)	0.297
Shell height (m)	0.270
D/H	1.10
Vessel Volume (m^3)	0.00513
Top head	Flat head
Maximum temp. (^{o}C)	25

3.7 Piping and Layout Diagram

The piping and layout diagram is presented in Figure 3.9. This layout was designed in such a way that mixture of methanol and sodium hydroxide flows by gravity in to the reactor and the condensed methanol also flows by gravity to the mixer.

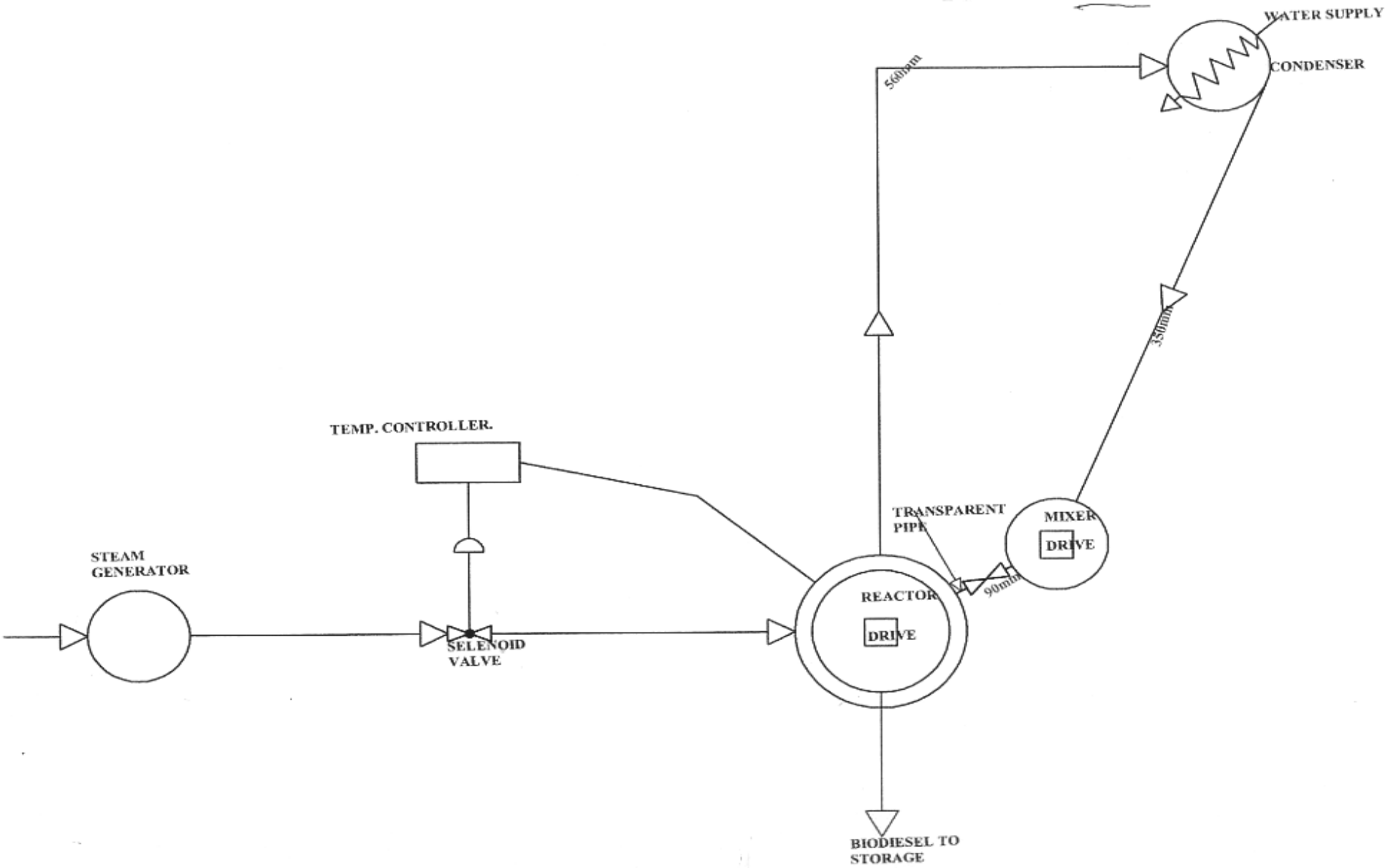


Figure 3.9: Piping and Layout Diagram of the Pilot Plant.

3.8 Instrumentation and Controls.

The governing controller for the transesterification reactor is an automatic control temperature system that controls the valve, which directs the flow of steam into the jacket. The flow of the reactants, products in and out of the transesterification reactor and cooling water to the condenser were controlled manually. In order to monitor the temperature of the vessel, a transmitter and a controller were implemented to maintain temperature and pressure set-points. The transesterification reactor is also equipped with sensor linked with controller to control the steam in/condensate out stream into and out of a jacket fitted around the vessel to maintain the vessel at the set-point temperature. For the stream that carries condensate from heating jacket, steam trap is installed to prevent heat and energy losses from expelling steam. The above mentioned type of instrumentation were employed due to its geometry, simplicity, safety provision, and cost effectiveness.

3.9 Construction and Installation

The construction of the biodiesel pilot plant was done in Nigeria at Haniga Nig. Ltd, Kaduna, under very close supervision of the designer. This was achieved using locally sourced materials. The individual equipment constructed were the Transesterificatin reactor, Mixer, and Condenser. These equipment were then tested for leakages. To achieve this, the equipment were covered with detergent solution, pressurized air was then passed through and observations made for leakages. These units after being successfully constructed and tested were coupled together and transported to the

Department of Chemical Engineering, Ahmadu Bello University, Zaria. The plant is shown coupled together in Figure 3.10. The pilot plant was then commissioned.



Figure 3.10: Picture of Biodiesel Pilot Plant

CHAPTER FOUR

PLANT COMMISSIONING AND PRODUCT ANALYSIS

Plant commissioning was preceded by the so called pre-commissioning phase which consists of operations aimed towards ascertaining that all construction and installation activities (electrical and mechanical) have been carried out correctly and that there were no leakages in the pipings and connections. The actual commissioning was a series of operations which certify that the plant, as designed, constructed and installed, of operating according to design.

4.1 Pilot Plant Operation Procedure

The steps encountered in the operation of the pilot plant from the raw materials to the finished products, includes

- Charging the raw material
- Transesterification
- Separation of products and
- Purification of biodiesel.

4.1.1 Charging Raw Materials

Raw materials namely cottonseed oil, methanol and sodium hydroxide were measured based on requirement and charged into the transesterification reactor and the mixer. This was done batch wise before the pilot plant was operated.

4.1.2 Transesterification

After charging**,** the methanol and sodium hydroxide were allowed to flow into the reactor and the reaction time was noted. The plant was operated until the transesterification reaction was completed.

4.1.3 Separation of Products.

After each batch operation the products obtained were allowed to settle by gravity for a period of eight hours, this was to enable very good separation. The top phase containing biodiesel was then decanted. The biodiesel obtained was distilled to remove excess methanol. The distillation was done using a rotary evaporator. The by-product, glycerol was also separated.

4.1.4 Purification of Biodiesel

The biodiesel obtained was washed properly using warm water and separated since the two phases are immiscible. Blowing dry air over the biodiesel vapourises any remaining moisture. The purified biodiesel was then characterized.

4.2 Start- Up And Shutdown Procedures

The commissioning of the pilot plant was done following the procedures below

4.2.1 Plant Start-up

The plant start-up involved the following steps.

(1) All equipment of the plant were ensured to be in good condition before proper connections were made.

(2) The steam generator and cooling water lines were started.

(3) Cottonseed oil was charged into the reactor.

(4) Methanol and alkali (earlier calculated) were also charged into the mixer and the stirrer started.

(5) Gradually the valve supplying methanol and the alkali was opened to allow flow into the reactor and time was noted.

(6) Finally the reactor stirrer was started and this indicated that full operation has began.

4.2.2 Shutdown Procedure

When the reaction time was achieved, the pilot plant was shutdown by the following;

(1) The reactor stirrer was stopped indicating that full operation had finished and time was noted.

(2) The valve supplying the methanol and sodium hydroxide into the reactor was.

(3) The steam generator and cooling waterui lines was stopped.

(4) All equipment of the plant were properly shutdown.

(5) Finally the reactor outlet valve was gradually opened to discharge the products.

4.3 Laboratory Analysis of Products

4.3.1 Procedure for an Automated Cetane Number Determination

The procedure for determining the cetane number of a fuel was by combustion of a sample in a constant volume combustion chamber filled with compressed air into which the fuel was injected via a temperature controlled injection nozzle. Means are provided for sensing the pressure in the combustion chamber and recording it for determining the cetane number there from. An automated sample supplier was provided to accommodate a plurality of fuel samples, including means for selectively connecting a pump to a particular sample and pressurizing it for injection into the combustion chamber and means for heating the combustion chamber and cooling the injector are provided for maintaining these devices at desired temperatures. This conforms to the procedure of ASTM D-2885

4.3.2 Procedure for the Determination of Kinematic Viscosity

Kinematic viscosity, ν, was measured by determining the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity, μ was obtained by multiplying the kinematic viscosity, ν, with the density, ρ, of the biodiesel. This conforms to the procedure of ASTM D6751-07a.

4.3.3 Procedure for the Determination of the Calorific Value

The gross calorific value was determined in a bomb calorimeter according to ASTM DIN 51900 in oxygen and at a pressure of 30 bars. To that end, a bomb was

inserted into a metal tank that can be assumed to be adiabatic. The tank was filled with water brought to a suitable temperature. The fuel to be investigated was input into the bomb, ignited by arcing, and combusted. The gross calorific value was determined by measuring the temperature of the bomb calorimeter.

4.3.4 Procedure for the Determination of the Flash Point

The determination of the flash point of fuel samples in temperature range from 40-360 ^{o}C by a manual Pensky-Martens closed-cup apparatus. This conforms to the procedure of ASTM D3828.

4.3.5 Procedure for the Determination of the IR Spectrum

The FT-IR spectrum was measured for the biodiesel samples. The procedure involved the following:

(1) Forming a spectrum library.

(2) Introducing a new basis and adding a new vector to the old basis.

(3) Individual analysis. Variances, error limits, and residual spectrums were also analysed. A windowing procedure was followed to reject spectrum portions where the transmittance approaches zero or which do not contain real information for some other reason. Since results of the biodiesel spectrum could not be compared in the library, an American standard chart was used to interpret the results as shown in Appendix II.

CHAPTER FIVE

RESULTS AND DISCUSSION

This chapter reports on the design results as well as data obtained from the test-runs carried out.

5.1 Design Results

The pilot plant design was carried out using Super Pro Chemical Engineering software, Hysis, and AutoCAD. The summary of the design results were already shown in Tables 3.10, 3.11, and 3.12 and the working drawings in Figures 3.4, 3.5, 3.6 and 3.7.

5.2 Physico-Chemical Properties of Biodiesel

The pilot plant was operated under different conditions and the products were obtained and analysed. The specific gravity, flash point, cetane number, viscosity, and calorific values of the biodiesel products for each of the runs were measured and the results are shown in Table 4.13. Four runs were carried out and the Physico-chemical properties determined.

Table 5.13: Properties of Biodiesel Produced.

Properties	**Physico-chemical Properties**				
	Sample 1 (0.75 hr)	**Sample 2** (1.0 hr)	**Sample 3** (1.5hr)	**Sample 4** (2.0 hr)	**Raw vegetable Oil**
Spec. gravity, (gm/ml)	0.87	0.88	0.90	0.91	0.92
Flash point (^oC)	137	141	122	117	89
*Cetane No	56	62	53	49	31
Viscosity (40^oC)	3.2	2.8	3.7	7.6	30.6
*Cal. Value kcal/kg	9210	9812	9235	9114	8302

Composition of methylesters (Biodiesel) was analysed using Fourier Transform Infrared Spectroscopy and its constituent concentration were quantified on percentage weight basis, using American Standard Chart for esters analysis American Standard Chart for Esters Analysis (1988). The results obtained were tabulated in Table 4.14.

Table 5.14: Methyl esters Concentration

Methyl esters	**Concentration (wt %)**				
	Sample 1 0.75 hr	**Sample 2** 1.0 hr	**Sample 3** 1.5 hr	**Sample 4** 2.0 hr	**Sample 5** 2.5 hr
Methyl arachidate	5.26	5.47	5.52	4.67	3.86
Methyl palmitate	4.50	3.60	1.07	2.32	2.57
Methyl stearate	3.41	2.83	1.41	1.15	1.13
Methyl Erucate	0.16	1.08	3.38	3.09	1.81
Methyl Linoteate	39.62	38.25	27.13	24.06	25.79
Methyl oleate	30.12	31.04	19.68	24.45	22.62
Methyl linolenate	3.86	3.95	8.92	2.95	2.34
Methyl myristate	0.56	1.47	5.73	3.48	3.02
Methyl laurate	-	1.52	3.89	1.29	-
Methyl palmitoleate	-	-	4.09	-	
Other Methyl esters	5.31	5.89	5.32	6.43	4.68
Total	92.80	95.02	86.14	73.89	67.82

5.3 Discussions

The biodiesel obtained was analysed in terms of both yield and physico-chemical properties. From Table 5.14, it can be seen that the content of the esters varies with batch reaction time and as time increases from 0.75hr to 1.0hr, the amount of the esters increases and decreases as the time elongates to 2.5hrs. Sample of 1 hr batch time provided biodiesel with maximum ester content of 95.02wt%. This is clearly shown in

Figure 5.11 which provides variation of batch reaction time with yields and selectivity of biodiesel.

5.3.1 Methyl esters Yield and Selectity

From the results of Table 5.15 the variation of yield against reaction time was plotted as shown in Figure 5.11. It is obvious that the yield of biodiesel depends on the batch time allowed in the transesterification reactor. The yield increased when batch time increased from 0.75 hour, reached a maximum at 1 hour and started to fall thereafter. The optimum batch time is therefore 1 hour. The fall in yield after 1 hour could be explained from the reversibility of the transesterification reaction. Also selectivity increased when the reaction time increased from 0.75 hour and reached a maximum at 1 hour. It started falling as time was elongated this is as a results of reversibility as shown in Table 5.15.

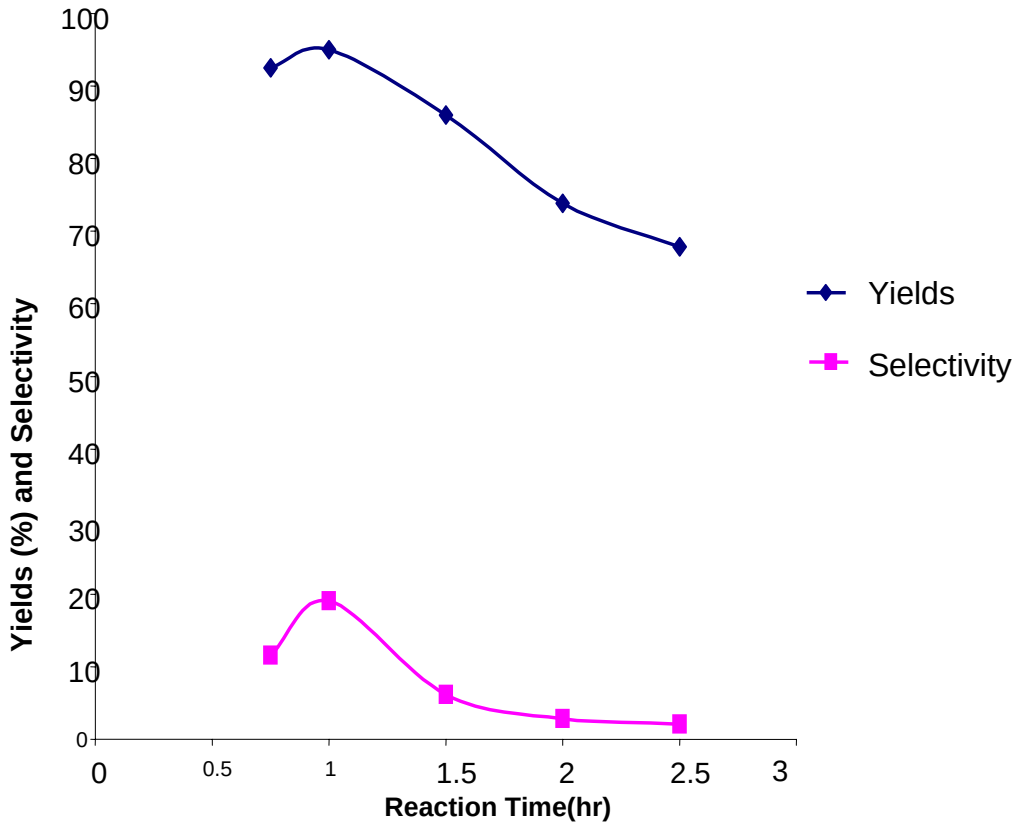


Figure 5.11: Variation of Yields (%) and Selectivity on Reaction Time

5.3.2 Physical Properties of Biodiesel

The American Society for Testing and Materials (ASTM) has given some guidelines with respect to the physico-chemical properties of biodiesel as contained in the ASTM D6751 Standards. All biodiesel produced in the US must meet up to this Standard. In the European Union, the Standard is EN14214. These Standards are summarised in Table 5.16. These properties were measured for our products and the results shown in

Table 5.13. The values obtained are compared with ASTM and EU standards in Table 5.16.

Table 5.16: Standards of Biodiesel Measurement (Gerhard, 2006).

PROPERTIES	**ASTM D6751**	**EN14214**
Flash point (^{o}C)	130.0 min.	120.0 min.
Viscosity (40^{o}C)(mm^{2}/s)	1.9-6.0	2.5-5.0
Cetane No	47 min	48 min
Spec.Gravity (gm/ml)	0.86-0.90	0.87-0.90
Cal. Value (kcal/kg)	Undefined	Undefined

5.3.2.1 Viscosities of Biodiesel

The high viscosity of the vegetable oil was the cause of severe operation problems, such as engine deposits [Plank and Lorber, 1992]. This is the major reason why vegetable oils have been largely abondaned as alternative to diesel fuel in favour of mono-alkyl esters. Hence, the lower the viscosity of the esters, the lesser the engine deposits and the better the fuel, as can be seen in the Table 5.13. Also increase in viscosities of biodiesel as time of reaction elongates could be attributed to the reversibility of the reaction leading to the formation of more unwanted products posing difficulty in gravity settling separation of the biodiesel from the mixture. The biodiesel produced has viscosity comparable to the ASTM D6751 and EN14214.

5.3.2.2 Cetane Value

Refiners and marketers measure the cetane of a fuel value to determine how readily it fuel ignites (Plank and Lorber, 1992). The higher the cetane numbers the better its ease of combustion. The values obtained for the biodiesel are well above both ASTM

D6751 and EN14214 minimum values. From Table 5.13 sample 2 provided fuel with the highest cetane number of 62 indicating good fuel which can readily ignite when injected into the combustion chamber. Fuels of this type also effectively get blended with lower quality diesel fuel to produce higher quality fuels with positive economic values (Plank and Lorber 1992).

5.3.2.3 Flash Point

It is the measure of a fuels' volatility and flammability. From Table 5.13 above, it can be seen that sample 2 provided biodiesel of highest flash point of 141^0C indicating much more safety in handling. The flash point 141 ^{o}C of the biodiesel produced was found to be within the limit of both ASTM and European Standards.

5.3.2.4 Calorific Value

This gives the amount of heat that could be generated when the fuel is burnt. Table 16 indicates variation in calorific values and the biodiesel with maximum – calorific value of 9812 kcal/kg signifies a fuel with the highest amount of energy. The higher the amount energy of a fuel, the better the fuel and the most economical it might be.

5.3.2.5 Specific Gravity

Table 16 also shows the specific gravities of the samples. Specific gravity indicates the density of the fuel which is the measure of the power output of a diesel engine per unit volume of fuel consumed. Research shows that biodiesel with specific gravity of 0.88 (gm/ml) provides lower particulates.

5.3.3 An Overview of the Results

Properties of the biodiesel produce fell within the ASTM and EU Standards D6751 and EN14214 respectively. Highest yield obtained was 95.02w/w%, and corresponded with reaction time of 1hour. This run also exhibited the best of fuel properties in terms of Specific gravity, Calorific value, Cetane number, Flash Point and Viscosity.

5.4 Economic Analysis

The pilot plant's economic viability was assessed and the details are contained in Appendix IV. The cost of the biodiesel product would depend on many factors, most critical of which is the cost of the vegetable oil used. If cheaper, non-edible oils can be sourced, the pilot plant can produce biodiesel at competitive costs, comparing well with AGO.

The economic analysis, assuming cost of raw materials to be 50% of the total cost, showed that the pilot plant has a DCFRR of 31.4%, ROR of 27.5% and a PBP of approximately 4 years. This makes the pilot plant a viable economic venture. Scale –up of the plant would indeed make it even more viable.

CHAPTER SIX

CONCLUSIONS AND RECOMMENDATIONS.

This chapter summarises the major conclusions that could be drawn from this work. Appropriate recommendations are also made.

6.1 Conclusions

From the results of this investigation, the following conclusions can be made.

1. The pilot plant was operated isothermally at 60^0C and at atmospheric pressure (1 bar) and an optimum batch reaction time of 1 hour was obtained. The product obtained at this batch time gave not only the highest yield of esters but the best of all physico-chemical properties tested.

2. Economic viability would depend on the cost of the raw material (vegetable oil). At an assumed raw material cost of 50% total production cost, the rate of return on investment was calculated to be 27.5%

3. This work has demonstrated the availability of local technological capacity in Nigeria for biodiesel production.

6.2 Recommendations

The following recommendations are made with respect to this work:

1. There is need to operate the pilot plant using other types of vegetable oils, especially the non-edible ones such as Castor and Jatropha oils.
2. A unit for separation of biodiesel from glycerin should be incorporated in the plant.
3. Once economic viability is well established, the pilot plant should be scaled up for commercial production.
4. Private sector participation should be encouraged in the scale-up of the plant.

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APPENDICES

APPENDIX I

DESIGN SIMULATION OF THE PILOT PLANT

This Appendix gives the results of the simulation studies of the pilot plant, showing the material and energy balance**s.**

Transesterification Reactor : Design, Reactions, Rating, Worksheet, Dynamics, Notes

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Conversion Reactor: Transesterification Reactor

CONNECTIONS

Inlet Stream Connections

Stream Name	From Unit Operation
Cottom Seed Oil	
Reactor Feed	MIX-100 Mixer

Outlet Stream Connections

Stream Name	To Unit Operation
Recy Methanol	RCY-1 Recycle

Raw Biodiesel

Energy Stream Connections

Stream Name	From Unit Operation
Reactor Duty	

PARAMETERS

Physical Parameters		Optional Heat Transfer:	
Delta P	Vessel Volume	Duty	Energy Stream
0.0000kPa	3.296e-002 m3	378.8 kJ/h	Reactor Duty

User Variables

REACTION DETAILS

Reaction: Rxn-1

Component	Mole Weight	Stoichiometric Coeff .
Cotton Seed oil	885.5	-1
Methanol	32.04	-3
Biodiesel	296.5	3

Glycerol	92.10	1

REACTION RESULTS FOR : Set-1

Extents

Name	Rank	Specified Conversion	Use Default	Actual % Conversion	Base Component	Reaction Extent (kgmole/h	% (
Rxn-1	0	98.00	Yes	98.00	Cotton Seed*	1.997e-002	

Balance

Components	Total Inflow kgmole/h)	Total Reaction (kgmole/h)	Total Outflow (kgmole/h	( )
Cotton Seed*	2.038e-002	-1.997e-002	4.076e-004	
Diesel*	0.0000	5.992e-002	5.992e-002	
Glycerol	0.0000	1.997e-002	1.997e-002	
NaOH	4.512e-003	0.0000	4.512e-003	
Methanol	0.1244	-5.992e-002	6.448e-002	

RATING

Sizing

Cylinder	Vertical	Reactor has a Boot: No
Volume 3.296e-002 m3	Diameter 0.3036 m	Height 0.4554

PROPERTIES

Cottom Seed Oil

	Overall	Liquid Phase
Vapour/Phase Fraction	0.0000	1.0000
Temperature: (C)	25.00	25.00
Pressure: (kPa)	101.3	101.3
Molar Flow (kgmole/h)	2.038e-002	2.038e-002
Mass Flow (kg/h)	18.05	18.05
Std Ideal Liq Vol Flow (L/h)	19.61	19.61
Molar Enthalpy (kJ/kgmole)	-1.693e+006	-1.693e+006
Mass Enthalpy (kJ/kg)	-1912	-1912
Molar Entropy (kJ/kgmole-C)	3910	3910

Mass Entropy (kJ/kg-C)	4.416	4.416
Heat Flow (kJ/h)	-3.451e+004	-3.451e+004
Molar Density (kgmole/m3)	1.034	1.034
Mass Density (kg/m3)	915.5	915.5
Std Ideal Liq Mass Density (kg/m3)	920.0	920.0
Liq Mass Density @Std Cond (kg/m3)	920.0	920.0
Molar Heat Capacity (kJ/kgmole-C)	1723	1723
Mass Heat Capacity (kJ/kg-C)	1.946	1.946
Thermal Conductivity (W/m-K)	0.1671	0.1671
Viscosity (cP)	7375	7375
Surface Tension (dyne/cm)	7.918	7.918
Molecular Weight	885.5	885.5
Z Factor	3.953e-002	3.953e-002

Reactor Feed

	Overall	Liquid Phase
Vapour/Phase Fraction	0.0000	1.0000

Temperature: (C)	25.00	25.00
Pressure: (kPa)	101.3	101.3
Molar Flow (kgmole/h)	0.1289	0.1289
Mass Flow (kg/h)	4.166	4.166
Std Ideal Liq Vol Flow (L/h)	5.111	5.111
Molar Enthalpy (kJ/kgmole)	-2.340e+005	-2.340e+005
Mass Enthalpy (kJ/kg)	-7239	-7239
Molar Entropy (kJ/kgmole-C)	42.61	42.61
Mass Entropy (kJ/kg-C)	1.318	1.318
Heat Flow (kJ/h)	-3.016e+004	-3.016e+004
Molar Density (kgmole/m3)	24.47	24.47
Mass Density (kg/m3)	791.0	791.0
Std Ideal Liq Mass Density (kg/m3)	815.3	815.3
Liq Mass Density @Std Cond (kg/m3)	801.2	801.2
Molar Heat Capacity (kJ/kgmole-C)	117.2	117.2
Mass Heat Capacity (kJ/kg-C)	3.626	3.626
Thermal Conductivity (W/m-K)	0.1848	0.1848

Viscosity (cP)	0.5259	0.5259
Surface Tension (dyne/cm)	2901	2901
Molecular Weight	32.32	32.32
Z Factor	1.670e-003	1.670e-003

Raw Biodiesel

	Overall	Liquid Phase	Vapour Phase
Vapour/Phase Fraction	0.0000	1.0000	0.0000
Temperature: (C)	60.00	60.00	60.00
Pressure: (kPa)	101.3	101.3	101.3
Molar Flow (kgmole/h)	0.1493	0.1493	0.0000
Mass Flow (kg/h)	22.21	22.21	0.0000
Std Ideal Liq Vol Flow (L/h)	24.88	24.88	0.0000
Molar Enthalpy (kJ/kgmole)	-4.357e+005	-4.357e+005	-1.997e+005
Mass Enthalpy (kJ/kg)	-2929	-2929	-6231
Molar Entropy (kJ/kgmole-C)	304.0	304.0	179.7
Mass Entropy (kJ/kg-C)	2.044	2.044	5.605
Heat Flow (kJ/h)	-6.505e+004	-6.505e+004	0.0000
Molar Density (kgmole/m3)	6.272	6.272	3.657e-002

Mass Density (kg/m3)	933.1	933.1	1.172
Std Ideal Liq Mass Density (kg/m3)	892.9	892.9	795.8
Liq Mass Density @Std Cond (kg/m3)	976.4	976.4	796.5
Molar Heat Capacity (kJ/kgmole-C)	322.9	322.9	46.63
Mass Heat Capacity (kJ/kg-C)	2.171	2.171	1.455
Thermal Conductivity (W/m-K)	0.1696	0.1696	1.563e-002
Viscosity (cP)	4.650	4.650	6.499e-003
Surface Tension (dyne/cm)	2409	2409	-
Molecular Weight	148.8	148.8	32.05
Z Factor	5.831e-003	5.831e-003	1.000

Recy Methanol

	Overall	Liquid Phase	Vapour Phase
Vapour/Phase Fraction	1.0000	0.0000	1.0000
Temperature: (C)	60.00	60.00	60.00
Pressure: (kPa)	101.3	101.3	101.3
Molar Flow (kgmole/h)	0.0000	0.0000	0.0000

Mass Flow (kg/h)	0.0000	0.0000	0.1030
Std Ideal Liq Vol Flow (L/h)	0.0000	0.0000	0.0000
Molar Enthalpy (kJ/kgmole)	-1.997e+005	-4.357e+005	-1.997e+005
Mass Enthalpy (kJ/kg)	-6231	-2929	-6231
Molar Entropy (kJ/kgmole-C)	179.7	304.0	179.7
Mass Entropy (kJ/kg-C)	5.605	2.044	5.605
Heat Flow (kJ/h)	0.0000	0.0000	0.0000
Molar Density (kgmole/m3)	3.657e-002	6.272	3.657e-002
Mass Density (kg/m3)	1.172	933.1	1.172
Std Ideal Liq Mass Density (kg/m3)	795.8	892.9	795.8
Liq Mass Density @Std Cond (kg/m3)	796.5	976.4	796.5
Molar Heat Capacity (kJ/kgmole-C)	46.63	322.9	46.63
Mass Heat Capacity (kJ/kg-C)	1.455	2.171	1.455
Thermal Conductivity (W/m-K)	1.563e-002	0.1696	1.563e-002
Viscosity (cP)	6.499e-003	4.650	6.499e-003
Surface Tension (dyne/cm)	---	2409	
Molecular Weight	32.05	148.8	32.05

Z Factor	1.000	5.831e-003	1.000

DYNAMICS

Vessel Parameters: Initialize from Product

Vessel Volume (m3)	3.296e-002	Level Calculator	Vertical cylinder
Vessel Diameter (m)	0.3036	Fraction Calculator	Use levels and nozzles
Vessel Height (m)	0.4554	Feed Delta P (kPa)	0.0000
Liquid Level Percent (%)	75.00	Vessel Pressure (kPa)	101.3

Holdup: Vessel Levels

Phase	Level (m)	Percent (%)	Volume (m^3)
Vapour	---	---	0.0000
Liquid	---	---	0.0000
Aqueous	---	---	0.0000

Holdup: Details

Phase	Accumulation	Moles	Volume

	kgmole/h)	(kgmole)	(m^3)
Vapour	0.0000	0.0000	0.0000
Liquid	0.0000	0.0000	0.0000
Aqueous	0.0000	0.0000	0.0000
Total	0.0000	0.0000	0.0000

Duty Valve Source : Direct_Q

Available UA (kJ/C-h)	3.600e+005	Heat Capacity (kJ/kgmole-C)	75.00
Utility HoldUp (kgmole)	100.0	Inlet Temp. (C)	15.00
Mole Flow (kgmole/h)	---	Outlet Temp. (C)	15.00
Min. Mole Flow (kgmole/h)	---	Temp. Approach (C)	10.00
Max. Mole Flow (kgmole/h)	---	Heat Flow (kJ/h)	378.8

Liquid Heater Height as % of Vessel Volume

Top of Heater : 5.00 % Bottom of Heater : 0.00 %

Heat Flow into the PFR: Cooling

Case (Main) (Workbook): All Pages

Workbook: Case (Main (

Material Streams

Name	Methanol	NaOH	Cottom Seed Oil	Reactor Feed	Raw Biodiesel
Vapour Fraction	0.0000	0.0000	0.0000	0.0000	0.0000
Temperature (C)	25.00	25.00	25.00	25.00	60.00
Pressure (kPa)	101.3	101.3	101.3	101.3	101.3
Molar Flow (kgmole/h)	0.1244	4.512e-003	2.038e-002	0.1289	0.1460
Mass Flow (kg/h)	3.986	0.1805	18.05	4.166	22.107
Liquid Volume Flow (L/h)	5.009	0.1012	19.61	5.111	24.75
Heat Flow (kJ/h)	-2.979e+004	-376.3	-3.451e+004	-3.016e+004	-6.505e+004
Name	Recy Methanol	Recycled Methanol			

Vapour Fraction	1.0000	0.0000
Temperature (C)	60.00	60.00
Pressure (kPa)	101.3	101.3
Molar Flow (kgmole/h)	0.0032	0.0000
Mass Flow (kg/h)	0.1030	0.0000
Liquid Volume Flow (L/h)	0.1297	0.0000
Heat Flow (kJ/h)	0.0000	0.0000

Compositions

Name	Methanol	NaOH	Cottom Seed Oil	Reactor Feed	Raw Biodiesel
Comp Mole Frac (Cotton Seed*)	0.0000	0.0000	1.0000	0.0000	0.0027
Comp Mole Frac (Diesel*)	0.0000	0.0000	0.0000	0.0000	0.4013
Comp Mole Frac (Glycerol)	0.0000	0.0000	0.0000	0.0000	0.1338
Comp Mole Frac (Methanol)	1.0000	0.0000	0.0000	0.9650	0.4319
Comp Mole Frac (NaOH)	0.0000	1.0000	0.0000	0.0350	0.0302

Name	Recy Methanol	Recycled Methanol
Comp Mole Frac (Cotton Seed*)	0.0000	0.0000
Comp Mole Frac (Diesel*)	0.0000	0.0000
Comp Mole Frac (Glycerol)	0.0000	0.0000

Comp Mole Frac (Methanol)	1.0000	1.0000
Comp Mole Frac (NaOH)	0.0000	0.0000

Energy Streams

Name	Reactor Duty
Heat Flow (kJ/h)	378.8

Unit Ops

Operation Name	Operation Type	Feeds	Products	Ignored	Calc. Level
MIX-100	Mixer	Methanol	Reactor Feed	No	500.0
		NaOH			
Transesterific Reactor	Conversion Reactor	Cottom Seed Oil	Raw Biodiesel	No	500.0
		Reactor Feed	Recy Methanol		
		Reactor Duty	Reactor Duty		
RCY-1	Recycle	Recy Methanol	Recycled Methanol	No	3500

APPENDIX II

Transesterification Reactor

Vessel Identification TK-1000-LIQUID-SOLID

1- Main Characteristics

1.1 Type .....Vertical
1.2 Operation.....Atmospheric
1.3 Useful Volume ……0.02472m³
1.4 Actual Volume……0.02474m³
1.5 Total Volume……0.03296m³
1.6 Volume Gap…….25%
1.7 Top Head..... Flat Head 10%
1.8 Bottom Head ..... Flat Head 10%

2-Geometry

2.1 Diameter……0.3036
2.2 Cylindrical Height……0.4554m
2.3 D/H ratio .....0.67.
24 Shell Height.....0.4554m

3- Materials

3.1 Top Head……Stainless Steel 304
3.2 Shell .....Stainless Steel 304
3.3 Bottom Head……..Stainless Steel 304

4- Product Main characteristic

4.1 Product Identification.....LIQUID-SOLID
4.2 Rheology .....Newtonian
43 Density 791.0kg/m³
4.4 Maximum Temperature 333.15K
4.5 Mixing Objectives .....Heat Transfer
.....Solids Dissolving
..... Fluid Motion

5- Agitator Characteristics

5.1 Position of agitator ..... Centered
5.2 Supporting.....With Footstool
5.3 Impellers Type..... Flat paddles
5.4 Number of Impellers.....6
5.5 Shaft Material……..Stainless Steel Type 304
5.6 Impellers Material……..Stainless Steel Type 304

6- Agitation System Characteristics

9.1 Reynolds Number.....53237
9.2 Aeration Number (N_A).....0.06929
9.3 Aeration Number Flooding(N_{FL}) 0.7644
9.4 Minimum Aeration Number (N_{LC}) 0.0429

9.5 Aeration Number Completely Dispersed (N_{CD}) 0.06929
9.6 Minimum Froud Number (N_{FRMin}) 0.075
9.7 Froud Number (N_{FR}) 0.5

7- Agitator Dimensions

7.1 Distance to Center..... 0 m
7.2 Impellers Diameter ..... 0.140 m
7.3 Blades Number..... 2
7.4 Blade Width 0.07993m
75 Blade Thickness 3.0000E-3m
7.6 Blade Angle 1.571rad
7.7 Hub Height 0.04296m
7.8 Hub Internal Diameter 0.01754m
7.9 Hub External Diameter 0.04296m
7.0 Hub Thickness..... 0.01271m
7.11 Disk Diameter..... 0.2098 m
7.12 Disk Thickness ..... 5.0000E-3 m
7.13 Shaft Length ……0.628 m
7.14 Shaft Diameter ..... 0.01754 m
7.15 Rotation 2.713Hz
7.16 Critical Speed ……..148.68 rpm
7.17 Calculated Power ……1025W
7.18 Adopted Power..... 1025W

Mixer Design Specifications

Vessel Identification..... **TK-1000-LIQUID-SOLID**

1- **Main Characteristics**

1.1	Type	Vertical
1.2	Operation	Atmospheric
1.3	Useful Volume	0.00513m³
1.4	Actual Volume	0.00513m³
1.5	Total Volume	0.005489m³
1.6	Volume Gap	7%
1.7	Top Head	Flat Head 10%
1.8	Bottom Head	Flat Head 10%

2-Geometry

2.1	Diameter	0.3000.m
2.2	Cylindrical Height	0.07765m
2.3	Shell Height	0.07765m

3- Materials

3.1	Top Head -	Stainless Steel 304
3.2	Shell	Stainless Steel 304
3.3	Bottom Head	Stainlees Steel 304

4- Product Main Characteristics

4.1	Product Identification	LIQUID-SOLID
42	Rheology	Newtonian
4.3	Density	879kg/m³
4.4	Maximum Temperature	298.15K

6-**Agitator Characteristics**

6.1	Position of agitator	Centered
6.2	Supporting	With Footstool
6.3	Impellers Type	Flat Radial paddles
6.4	Number of Impellers	1
6.5	Shaft Material	Stainless Steel
6.6	Impellers Material	StainlessSteel

7 - Impellers Position Regarding LTI

7.1	1st Impeller	0m

8 - **Agitation System Characteristics**

8.1	Reynolds Number	43337
8.2	Aeration Number (N_A)	0.06929
8.3	Aeration Number Flooding(N_{FL})	0.3644

8.4 Minimum Aeration Number (N_{LC}) 0.0329
8.5 Aeration Number Completely Dispersed (N_{CD}) 0.06929
8.6 Minimum Froud Number (N_{FRMin}) 0.045
87 Froud Number (N_{FR}) 0.3

9 - Agitator Dimensions

9.1 Distance to Center 0m
9.2 Impellers Diameter 0.120m
9.3 Blades Number 2
9.4 Blade Width 0.0517m
9.5 Blade Thickness 3.0000E-3m
9.6 Blade Angle 1.571rad
9.7 Hub Height 0.0320m
9.8 Hub Internal Diameter 0.01754m
9.9 Hub External Diameter 0.0320m
9.10 Hub Thickness 0.0106m
9.11 Disk Diameter 0.0876m
9.12 Disk Thickness 5.0000E-3m
9.13 Shaft Length 0.390m
9.14 Shaft Diameter 0.01754m
9.15 Rotation 0.94
9.16 Critical Speed 140rpm
9.17 Calculated Power 425W
9.18 Adopted Power 425W

Heat Exchanger Specification Sheet

1	Company: .						
2	Location:						
3	Service of Unit: Simple Condensation Verification Problem						
4	Date:						
5	Date:	Rev No.: Job No.:					
6	Size	120 / 470 mm	Type CEU	hor Connected in	1 Parallel	1	series
7	Surf/unit(eff.)	0.12 m2	Shells/unit 1		Surf/shell (eff.)	0.12	m2
8	**PERFORMANCE OF ONE UNIT**						
9	Fluid allocation		**Shell Side**		**Tube Side**		
10	Fluid name		Water		Methanol vapours		
11	Fluid quantity, Total	kg/hr	7.20		0.1030		
12	Vapor (In/Out)	kg/hr	7.20				
13	Liquid	kg/hr		7.20	0.1030	0.1030	
14	Noncondensable	kg/hr					
15							
16	Temperature (In/Out)	C	20	25	60	25	
17	Dew / Bubble point	C					
18	Density	kg/m3	998.92	998.15	1.172	933.1	
19	Viscosity	mPa*s					
20	Molecular wt, Vap						
21	Molecular wt, NC						
22	Specific heat	kJ/(kg*K)	4.195	4.192	1.946	1.946	
23	Thermal conductivity	W/(m*K)	0.587	0.6	1.563e-003	0.1696	
24	Latent heat	kJ/kg			-6231	-2929	
25	Pressure	bar	1		1		
26	Velocity	m/s	0.5		0.28		
27	Pressure drop, allow./calc.	bar	0.1	0	0.1	0.005	
28	Fouling resist. (min)	m2*K/W	0.00004		0.00005		
29	Heat exchanged	4994 W			MTD corrected	16.03	C
30	Transfer rate, Service	667.7	Dirty	883.3	Clean	965.1	
31	**CONSTRUCTION OF ONE SHELL**				**Sketch**		
32			**Shell Side**	**Tube Side**			
33	Design/Test pressure	bar	1.0 / Code	1.0 / Code			
34	Design temperature	C	20	60			
35	Number passes per shell		1	8			
36	Corrosion allowance	mm	1.59	1.59			
37	Connections	In	8.467/ 50ANSI	8.4674 / 100 ANSI			
38	Size/rating	Out	8.467/ 50 ANSI	8.467 / 100ANSI			
39	mm	Intermediate	/ 50 ANSI	/ 100 ANSI			
40	Tube No. 4Us	OD 6.35	Tks-avg 0.413 mm	Length470 mm	Pitch	7.934	mm
41	Tube type		Material	SS304	Tube pattern	10	
42	Shell CS	ID	OD 120 mm	Shell cover	CS		
43	Channel or bonnet	CS		Channel cover	CS		
44	Tubesheet-stationary	CS		Tubesheet-floating			
45	Floating head cover			Impingement protection	Circular plate on bundle		
46	Baffle-crossing CS	Type Single seg	Cut (%d) 11.4	vert	Spacing: c/c		mm
47	Baffle-long	Seal type			Inlet	120	mm
48	Supports-tube	U-bend		Type			
49	Bypass seal		Tube-tubesheet joint		Expand		
50	Expansion joint		Type				
51	RhoV2-Inlet nozzle	8	Bundle entrance	0	Bundle exit	0	kg/(m*s2)
52	Gaskets - Shell side		Tube Side				
53	Floating head						
54	Code requirements	ASME Code Sec VIII Div 1		TEMA class	R		
55	Weight/Shell	134.7	Filled with water	152.6	Bundle	5.83	kg
56	Remarks						

Thermal Details - General

		Shell Side		Tube Side	
Liquids (in/out)	**kg/hr**		7.20	0.1030	0.1030
Temperature (in/out)	**C**	20	25	60	25
Dew point or bubble point	**C**				
Film coefficient	**W/(m2*K)**	2127.53		484.8	
Fouling resistance	**m2*K/W**	0.00004		0.00005	
Velocity	**m/s**	0.5		0.28	
Pressure drop (allow./calc.)	**bar**	0.1	0	0.1	0.005

Total heat exchanged	**W**	1664.67	Type	CEU hor 1ser 1 par
Overall coef. - dirty	**W/(m2*K)**	294.43	Shell size	120 / 470 **mm**
Effective surface area	**m2**	0.5	Tube No-OD	8 / 6.35 **mm**
MTD corrected	**C**	5.34	Baffles	single seg14.67%vert
MTD correction factor		1	Tube passes	8

Thermal Details - Thermal Resistances

		Clean	Specified Foul	Max. Foul
Area reqd.	**m2**	0.1	0.12	0.3
Excess surface		14.85	10.76	
Overall coefficient	**W/(m2*K)**	965.1	294.43	222.57
Overall resistance	**m2*K/W**	0.00104	0.00113	0.0015
Shell side fouling	**m2*K/W**		0.00004	0.00017
Tube side fouling (at tube ID)			0.00005	0.00025
Distribution of overall resistance				
Shell side film	**%**	5.04	4.613	3.487
Shell side fouling	**%**		1.11	4.3
Tube wall	**%**	2.85	2.607	1.97
Tube side fouling	**%**		0.597	6.507
Tube side film	**%**	25.44	23.287	17.603

Thermal Details - Coefficients

		Shell Side	Tube Side
Film coefficients	**W/(m2*K)**		
As calculated by program		2127.53	484.8
As specified by user in input			
User specified multiplier		1	1
As used in design		3191.3	745.2
Desuperheating coefficient			
Condensing coefficient		9646	
Vapor sensible coefficient			
Liquid sensible coefficient			745.2
Boiling coefficient			

Liquid cooling coefficient		307.8	
Reynolds number		14.91	5498.73
Fin efficiency factor		1	
Mean metal temperature	**C**	21.67	26.07

Thermal Details - MTD and Flux

Mean temperature difference	**C**	**Flux**	**W/m2**
Corrected MTD used in design	5.34	Actual flux	3567.67
LMTD		Boiling fluid	
MTD correction factor	1	Nucleate maximum flux	
Long. baffle efficiency factor		Program maximum flux	
Calculated corrected MTD	5.34	Specified maximum flux	
User specified corrected MTD		Controlling maximum flux	
Flow direction			

Thermal Details - Pressure Drop

	Shell Side		**Tube Side**	
Press Drop **bar**				
Allowable	0.1		0.1	
Calculated, clean	0		0.005	
Calculated, dirty	0		0.005	
User specified bundle multiplier	1		1	
Velocity and pressure drop distribution	**m/s**	**%dp**	**m/s**	**%dp**
Inlet nozzle	2.016	31.56	0.08	0.84
Entering bundle	0.063	0.306	0.093	4.65
Crossflow	0.5	2.49		
Through baffle windows	0.143	0.627		
Through tubes			0.093	22.347
Exiting bundle	0		0.093	4.657
Outlet nozzle	0	0.02	0.08	1.26

Thermal Details - Shell Side Flow

Shell Side Stream Analysis	**Flow Fraction**	**Diametric Clearances mm**
Crossflow	9.6	
Clearance: Baffle hole - tube OD	1.67	0.0877
Clearance: Shell ID - baffle OD	1.663	1.06
Clearance: Shell ID - bundle OTL	17.06	6.35
Rho*V2 Analysis	**Rho*V2 kg/(m*s2)**	**TEMA Limit kg/(m*s2)**

Mechanical Details - Shell & Heads Nozzles

TEMA Type CEU		**Shell**	**Front Head**
Cylinder outside diameter	**mm**	168.28	168.28
Cylinder inside diameter	**mm**	154.05	154.05
Kettle outside diameter	**mm**		
Vapor belt outside diameter	**mm**		
Vapor belt length	**mm**		
Nozzles - nominal OD		**Shell Side**	**Tube Side**
Inlet nozzle	**mm**	25.4 / 1	25.4 / 1
Outlet nozzle	**mm**	25.4 / 1	25.4 / 1
Drain	**mm**	/ 1	/
Inlet dome	**mm**		
Distributor belt	**mm**		

Arrangement - total shells: 1 Connected in 1 parallel 1 series

Total price Dollar(US) 3540

Mechanical Details - Bundle

Baffle type	single seg			Clearance: Shell id - bundle otl	19.05
Inlet spacing	**mm**	169.8		Clearance: Baffle hole - tube od	0.79
C-C spacing	**mm**	50.8		Clearance: Shell id - baffle od	3.18
Outlet spacing	**mm**	195.2		Clearance: Baffle od - bundle otl	15.88
Number of baffles		12			
Supports		1		Impingement protection	Circular plate on bundle
Baffle cut		44	vert	Sealing strips (pairs)	1
Multi-segmental inner cut			%	Outer tube limit	135
Multi-segmental outer cut			%	Open distance at top	46.83
Baffle thickness	**mm**	3.18		Open distance at bottom	29.43

Mechanical Details - Tubes

Tube length	**mm**	1000		Tube o.d.	**mm**	19.05
Number of tubes		8		Tube wall thickness	**mm**	1.24
Tube pitch	**mm**	23.81		Tube wall specification		avg
Tube pattern				Tube type		
Tube passes				Fin height	**mm**	
Tube pass layout		Quadrant		Fin thickness	**mm**	
Tubesheet thickness (est.)	**mm**	25.4		Fin density	**#/m**	
Tube-tubesheet joint	expand			Area ratio Ao/Ai		1.15
Pass partition lane width	**mm**	15.88		Twisted tape insert width	**mm**	
Deviation in tubes/pass		5	%	Twist ratio		

Mechanical Details - Vibration Analysis

For liquids and gases	**Inlet**	**Bundle**	**Outlet**	**U-bend**
Vibration Indication	no	no	no	no

Tube unsupported span		**mm**	220.6	101.6	246	
Crossflow velocity	v	**m/s**	0.19	0.5	0	
Critical velocity	vc	**m/s**	538.82	3083.55	22.65	
Crossflow/critical velocity Vibration indicated if > 1.0	v/vc		0	0	0	
Tube natural frequency	fn	**Hz**	856.6	4038.5	838.5	
Vortex shedding amplitude (limit 0.4)		**mm**				
Turbulent buffeting amplitude (limit 0.4)		**mm**	0	0	0	

Mechanical Details - Acoustic Analysis

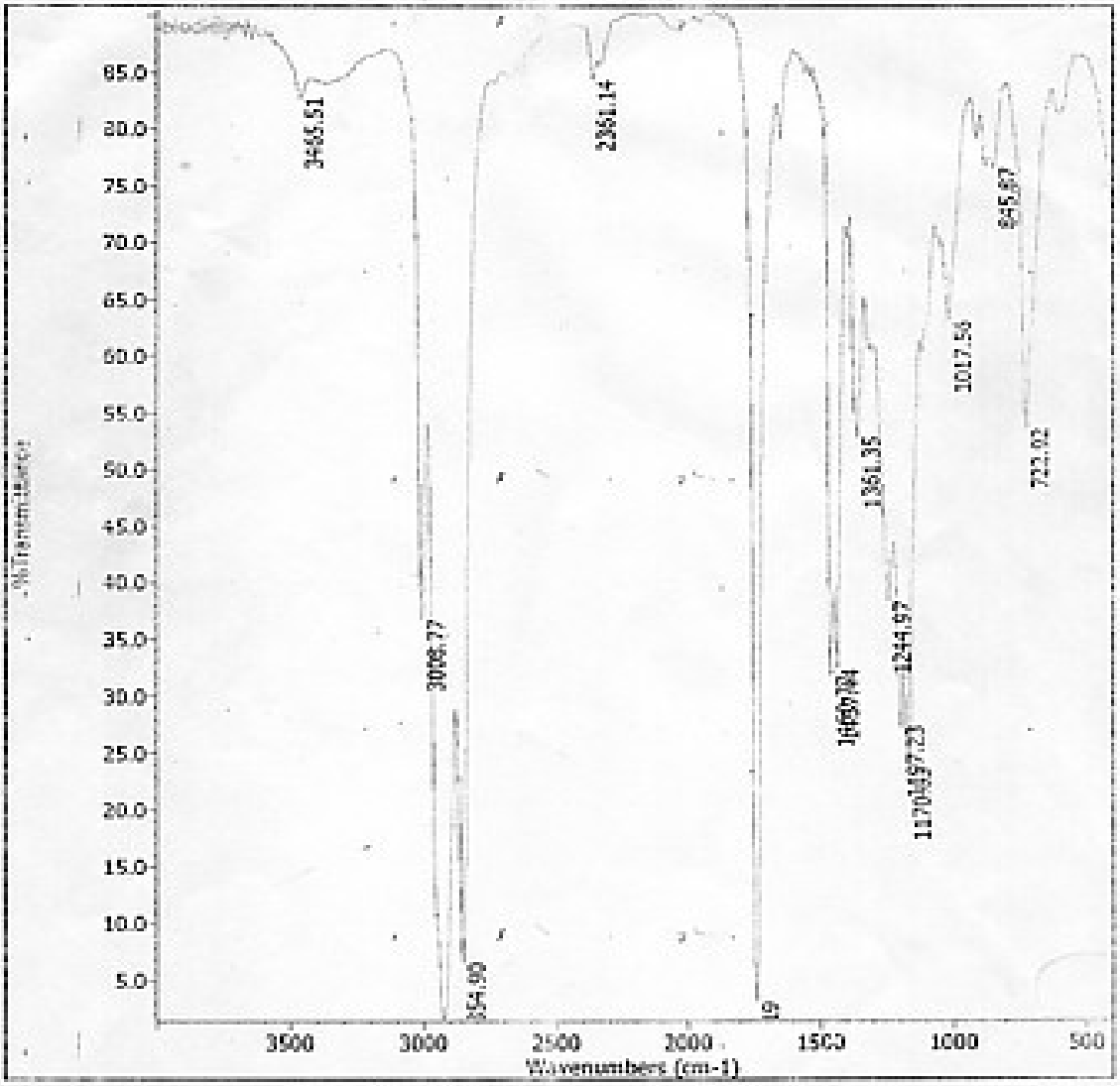
For gases only			**Inlet**	**Bundle**	**Outlet**	**U-bend**
Acoustic resonance indication			no	no	no	no
Tube unsupported span	l	**mm**	220.6	101.6	246	
Crossflow velocity	v	**m/s**	0.19	0.5	0	
Shell acoustic frequency	fa	**Hz**	979.4			
Vortex shedding frequency	fs	**Hz**	2.2	5.8		
Turbulent buffeting freq.	ftb	**Hz**	2.9			
Condition "a" ratio	fa/fs		441.49			
	fa/ftb		332.73			
Resonance indicated if 0.8 - 1.2						
Condition "b" velocity		**m/s**	21.74	21.74		
Resonance indicated if v > "b" velocity						
Condition "c" velocity		**m/s**	83.33	83.33		
Condition "c" -			219			
Resonance indicated if v > "c" veloicty and Condition "c" > 2000						

IR SPECTROSCOPY RESULTS

Peak finding results for: biodiesel
Frequency: 400.00 – 4000.00, threshold: 97.274, sensitivity: 50.00
Peak finding result table:

Peak#	1	2	3	4	5	6	7	8	9
Position	3465.51	3008.77	2926.14	2854.80	2361.14	1743.19	1460.77	1437.44	1361.35
Height	82.734	30.700	1.330	6.476	84.404	2.882	31.731	32.275	52.881

Peak#	10	11	12	13	14	15			
Position	1244.97	1197.23	1170.03	1017.56	845.87	722.02			
Height	38.232	27.198	23.527	62.944	76.554	53.531			

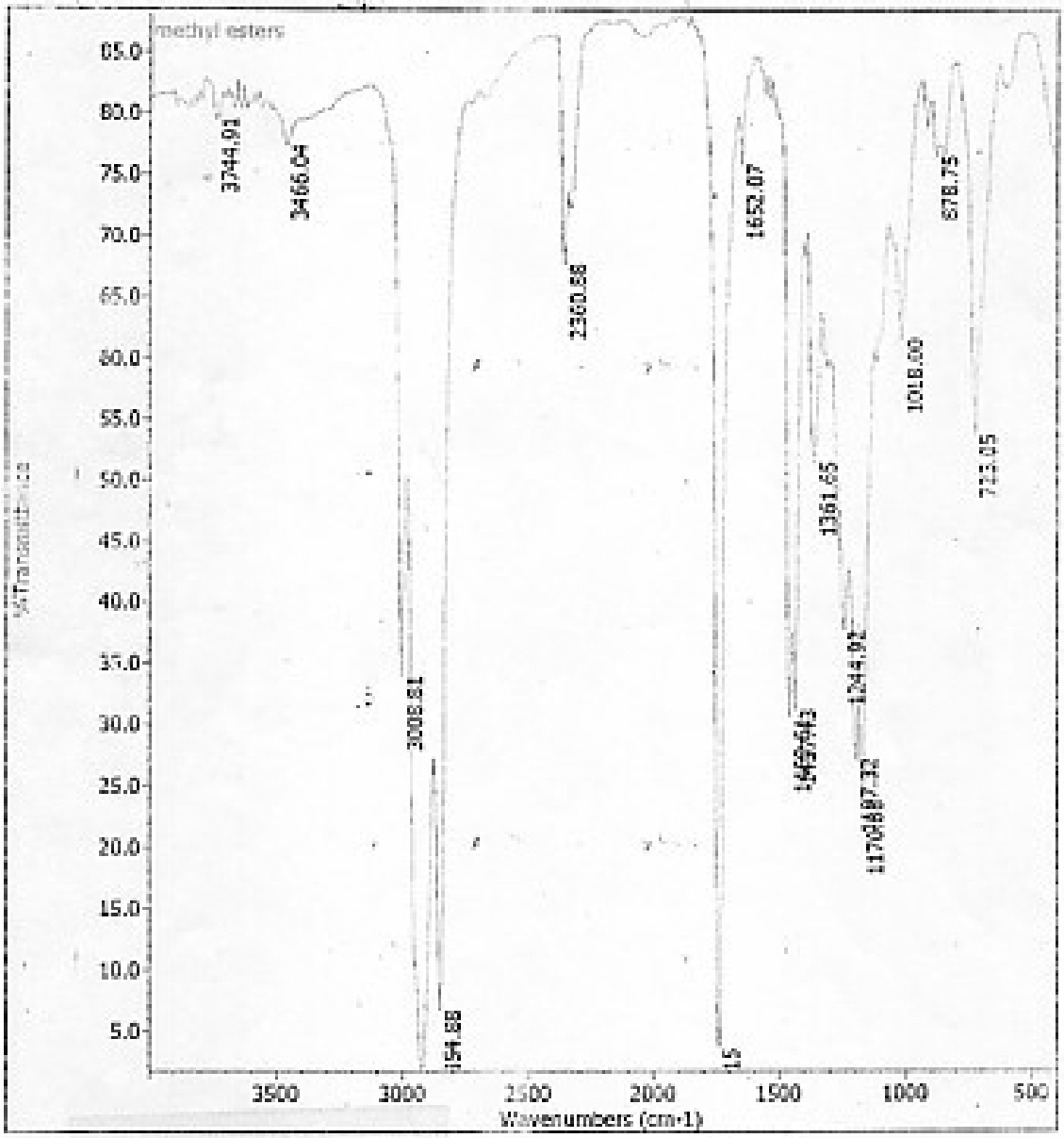


Sample Time 45 min.

Peak finding results for: methyl esters 2
Frequency: 400.00 - 3999.64, threshold: 87.531, sensitivity: 50.00
Peak finding result table:

Peak#	1	2	3	4	5	6	7	8	9
Position	3744.91	3466.04	3008.81	2925.92	2854.88	2360.88	1743.15	1652.07	1460.61
Height	79.481	77.331	33.061	1.646	6.571	67.672	3.361	75.818	30.437

Peak#	10	11	12	13	14	15	16	17	
Position	1437.43	1361.65	1244.92	1187.32	1170.88	1018.00	878.75	723.05	
Height	31.076	51.412	37.558	27.003	23.640	61.555	76.498	53.660	



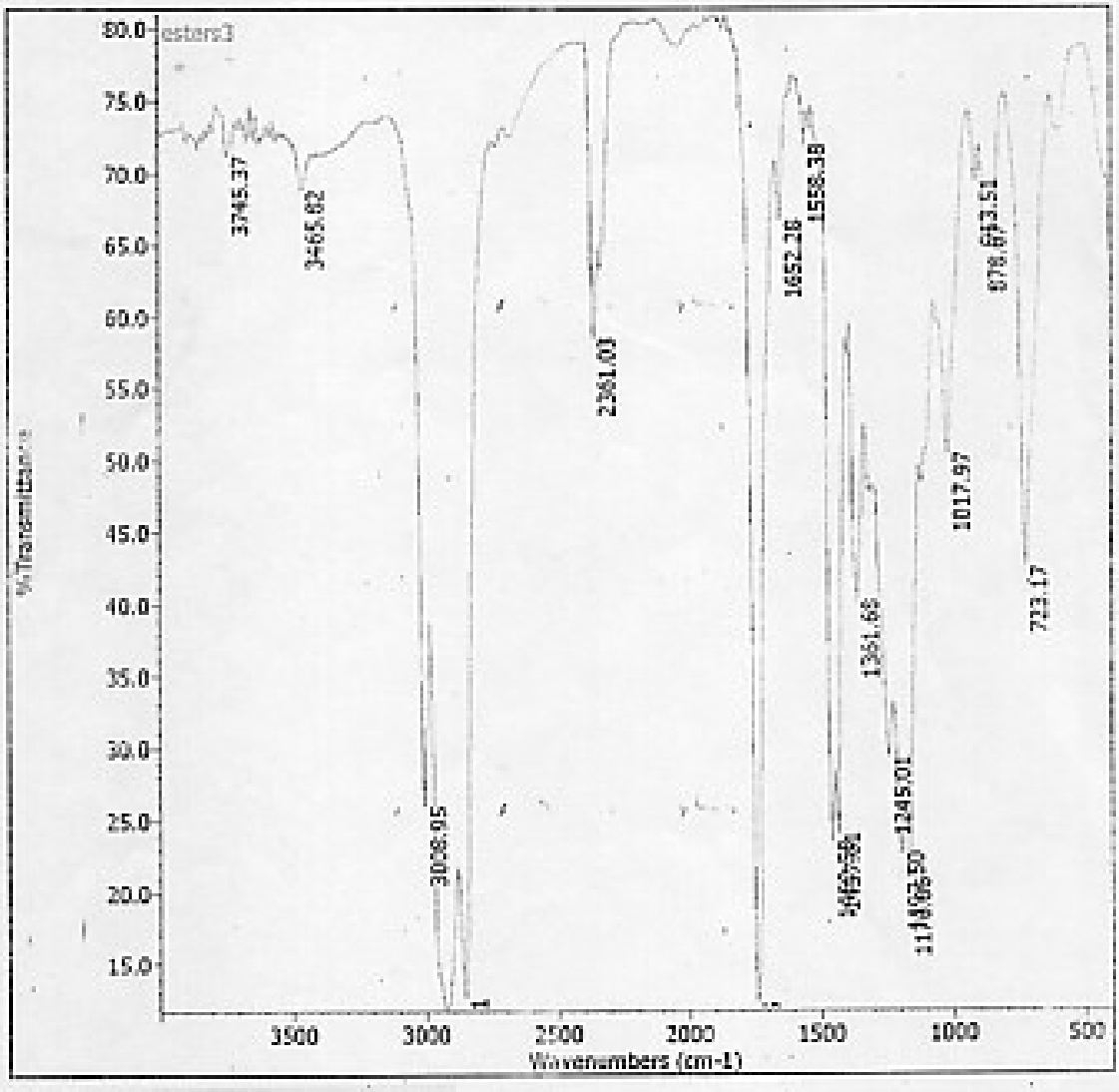
Sample Time 1 hr.

Peak finding results for: esters3
Frequency: 400.00 - 3999.64, threshold: 81.005, sensitivity: 50.00
Peak finding result table:

Peak#	1	2	3	4	5	6	7	8	9
Position	3745.37	3465.82	3008.95	2924.91	2854.71	2361.03	1743.15	1652.28	1558.38
Height	71.139	68.800	25.856	11.667	12.515	50.385	12.115	68.640	71.808

Peak#	10	11	12	13	14	15	16	17	18
Position	1460.58	1437.61	1381.68	1245.01	1197.50	1170.86	1017.97	913.51	878.67
Height	23.344	23.872	40.112	29.157	22.474	20.699	50.265	69.315	66.182

Peak#	19
Position	723.17
Height	42.369

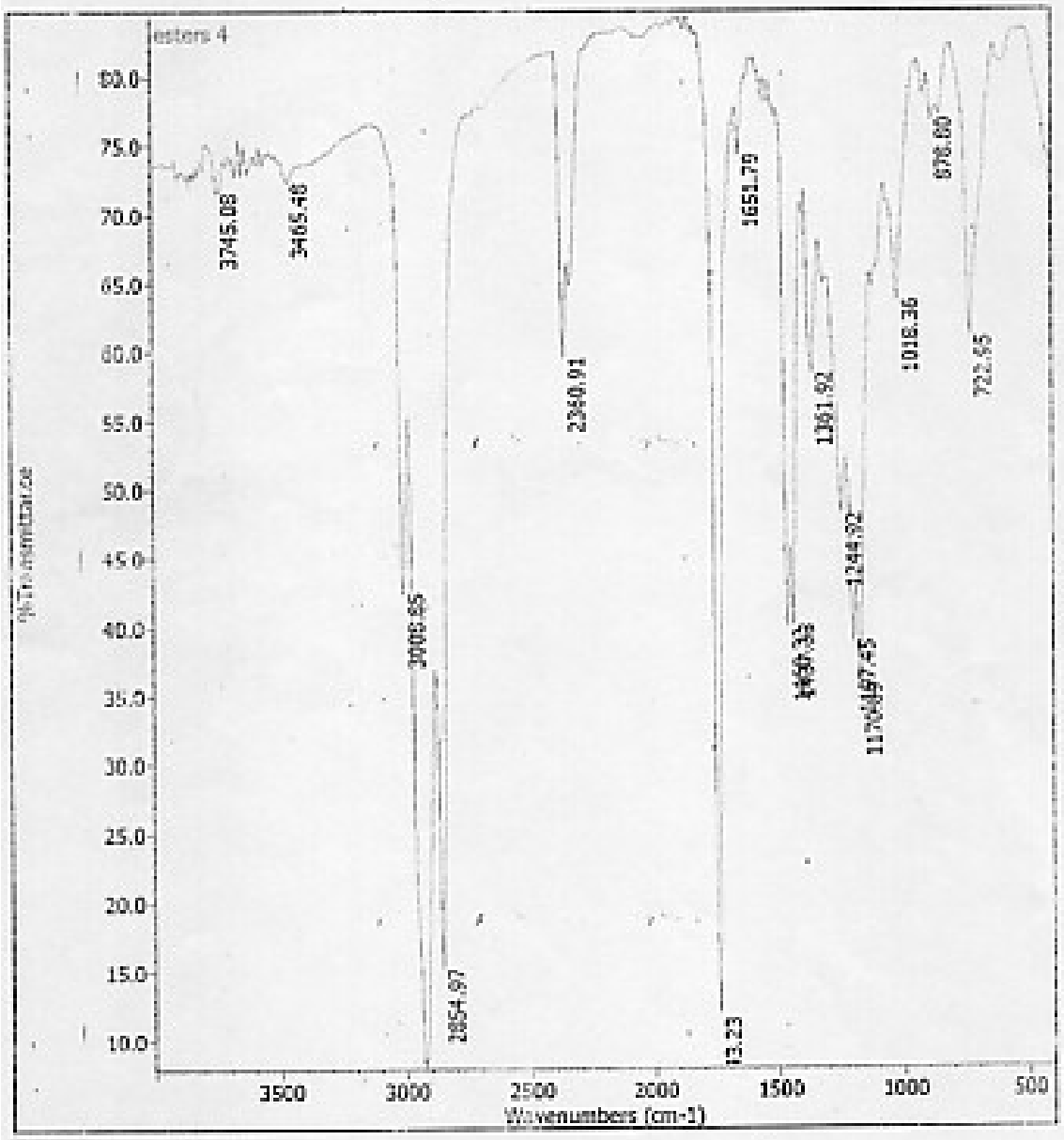


Sample Time 1hr 30min.

Peak finding results for: esters 4
Frequency: 400.00 - 3999.64, threshold: 91.005, sensitivity: 50.00
Peak finding result table:

Peak#	1	2	3	4	5	6	7	8	9
Position	3745.08	3465.48	3008.85	2926.10	2854.97	2360.91	1743.23	1651.79	1460.32
Height	71.522	72.295	42.344	7.769	15.104	59.476	11.338	74.250	39.709

Peak#	10	11	12	13	14	15	16	17	
Position	1437.33	1361.92	1244.92	1197.45	1170.80	1018.36	870.80	722.95	
Height	39.957	58.315	48.049	38.756	35.656	63.627	76.815	60.921	

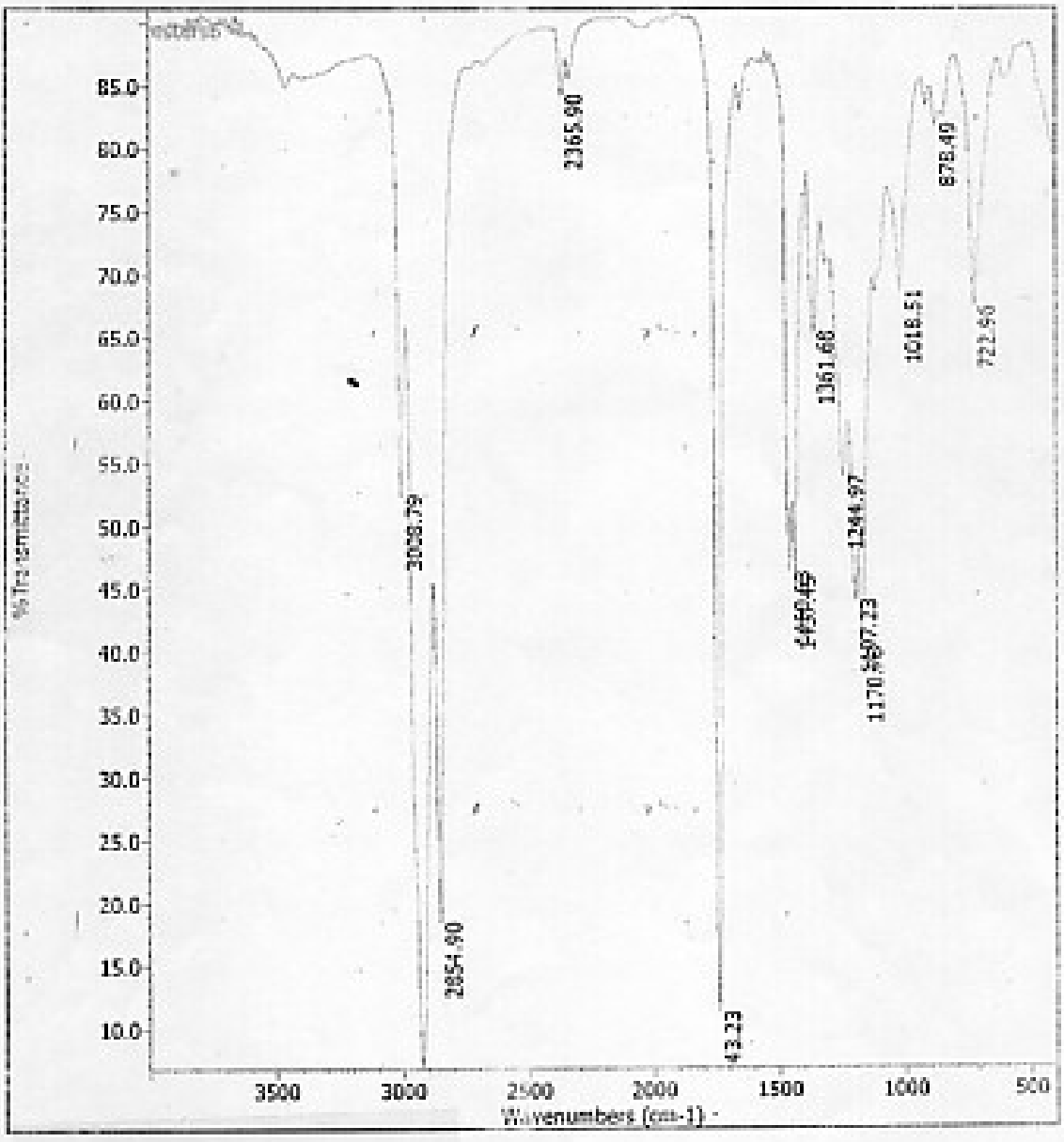


Sample Time 2 hrs.

Peak finding results for: esters5
Frequency: 400.00 - 3999.64, threshold: 87.531, sensitivity: 50.00
Peak finding result table:

Peak#	1	2	3	4	5	6	7	8	9
Position	3008.79	2926.18	2854.90	2365.00	1743.23	1460.63	1437.45	1361.68	1244.97
Height	52.300	6.710	10.216	84.288	11.083	46.153	45.841	65.357	53.835

Peak#	10	11	12	13	14
Position	1197.23	1170.78	1018.51	878.49	722.96
Height	43.828	40.020	68.621	81.930	67.356



Sample Time 2 hrs 30min.

APPENDIX III

ECONOMIC ANALYSIS

Economic analysis of the pilot plant is necessary in order to assess viability though the production capacity of the pilot plant is much lower, the analysis will guide a possible scale-up. Biodiesel has to compete with AGO and its economic competitiveness would depend on the price of crude oil in the international market and to some extent the local price of AGO in the Nigerian market (Udeh, and Keth, 2001).

Calculation of total investment cost

Purchased equipment cost

The total equipment capital cost (PEC) was estimated to be ₦400, 000 based on the actual cost of construction of the pilot plant by Haniga (Nigeria) Ltd.

Installation cost

The installation cost (IC) was found to be 35-45 % of the purchased equipment cost (PEC) [Udeh, 2001]. Now, taking the installation cost to be 40 % of the purchased equipment (PEC), then

IC = 40%.PEC

IC = ₦160, 000

Instrumentation and control installation cost

The Instrumentation and control installation cost was found to be 6-30 % of the purchased equipment cost (PEC). Now taking the Instrumentation and control installation cost (IC) to be 15 % of the purchased equipment cost (PEC), then

ICIC = 15 % PEC.

ICIC = ₦80, 000

Piping installation cost

The Piping installation cost (PIC) was found to be 10-80 % of the purchased equipment cost (PEC). Now taking Piping installation cost (PIC) to be 15 % of the purchased equipment cost (PEC), then

PIC = 15 %PEC

PIC = ₦60, 000

Electrical installation cost

The electrical installation cost (EIC) was found to be 10-40 % of the purchased equipment cost (PEC). Now taking the electrical installation cost (IC), then

EIC = 15 % PEC.

EIC = ₦60, 000

Building process and auxiliary cost

The Building process and auxiliary cost (BPAC) was found to be 10-70 % of the purchased equipment cost (PEC). Now taking the Building process and auxiliary cost to be 20 % of the purchased equipment cost then,

BPAC = 20% PEC

BPAC = ₦80, 000

Service facility cost.

The service facility cost (SFC) was found to be 30-80 % of the purchased equipment cost (PEC). Now taking the Service facility cost (SFC) to be 40 % of the purchased equipment cost (PEC).

SFC = 40 % PEC

SFC = ₦160, 000

Yard improvement cost

The yard improvement cost (YIC) was found to be 10-50 % of the purchased equipment cost (PEC). Now taking improvement cost (YIC) to be 20% of the purchased equipment cost (PEC), then

YIC = 20 %PEC

YIC = ₦60, 000

Cost of land.

The cost of land (LC) was found to be 4-8 % of the purchased equipment cost (PEC).Now taking cost of land to be 5% of the purchased equipment cost (PEC), then

LC = 5% PEC

LC = ₦20, 000

All the cost calculated so far is referred as the direct costs. So, the total total direct cost can be computed as the sum of all the various costs above. That is, denoting total direct by TDC.

TDC = PEC+ IC+ICIC+PIC+EIC+BPAC+SFC+YIC+LC

TDC = ₦1, 080,000

Indirect cost

The indirect cost, which are the cost that are neither directly involved in the material and labour of installation nor in the complete facility. They include the costs calculated below.

Engineering and supervision cost

The engineering and supervision cost (ESC) was found to be 5-30 % of the total direct cost (TDC). Now, taking the engineering and supervision cost (ESC) to be 15% of the total direct cost (TDC), then

ESC = 10 % TDC

ESC = ₦108, 000

Construction expenses

The Construction expenses CE) were found to be 10 % of the total direct cost (TDC). Now, this means that the Construction expenses (CE) is 10 % of the total direct (TDC) then

CE = 10% TDC

CE = ₦108, 000

Contractors' fee

The Contractors' fee (CF) was found to be 2-7% of the total direct cost (TDC). Now that

The Contractors' fee (CF) to be 4% of the total direct cost (TDC), then

CF = 4% TDC

CF = ₦43, 200

Contingency fee

The Contingency fee (COF) was found to be 8-20% of the total direct cost (TDC). Now, taking the Contingency fee (COF) to be 15% of the total direct cost (TDC), then

COF = 15% TDC

COF = ₦118, 000

All the cost after direct costs are known to be part of the indirect cost. The total indirect is thus given as

TIC = ESC + CE + CF + COF

TIC = ₦ 378, 000

Fixed capital investment

Fixed capital investment is given as the sum of the direct and indirect costs. That is, denoting the fixed capital investment by FCI.

FCI = TDC + TIC

FCI = ₦1, 458, 000

Working capital investment cost

The Working capital investment cost was found to be 10-20% of the fixed capital investment (FCI). Now taking the Working capital cost (WCIC) is 15% of fixed capital investment cost (FCI), then

WCIC = 15% FCI

WCIC = ₦ 218, 700

Total capital investment

The total capital investment is given as the sum of the fixed capital and Working capital investments

TCI = FCI + WCIC

TCI = ₦1, 676, 000

Estimation of total production cost (TPC)

Fixed Charges

Depreciation cost

The depreciation (Dep) was found to be 10% of the fixed capital investment (FCI). Now taking the depreciation (Dep) to be 10% of the fixed capital investment (FCI), then

Dep = 10% FCI

Dep = ₦145, 800

Local Taxes

The local taxes (LT) were found to be 1-2% of the fixed capital investment (FCI). Now taking the local taxes (LT) to be 1.5% of the fxed capital investment (FCI), them

LT= 1.5% FCI

LT = ₦21, 870

Insurance

The insurance (Ins) was found to be 0.4-1% of the fixed capital investment (FCI). Now, taking the insurance (Ins) to be 0.7% of the fixed capital investment (FCI). Then

Ins = 0.7% FCI

Ins = ₦10, 206

Rent

The rent (Rent) was found to be 8-12% of the fixed capital investment (FCI). Now taking the rent to be 9% of the fixed capital investment (FCI). Then

Rent = 9% FCI

Rent = ₦131, 220

Therefore, the total production cost which is the sum of the depreciation, local taxes, insurance and rent is equal to

TPC = Dep + LT+ Ins + Rent

TPC = ₦ 309, 076

Direct production costs

Raw material

The cost of raw material (RMC) was found to be 10-50% of the total production cost (TPC). Now taking the cost of the raw material (RMC) to be 50% of the total production cost (TPC), then

RMC = 50% TPC

RMC = ₦154, 538

Operating labour

The cost of operating labour (OLC) was found to be 10-20% of the total production cost (TPC). Now, taking the of operating labour (OLC) to be 17% of the total production cost (TPC), then

OLC = 17% TPC

OLC = ₦52, 542.92

Direct supervisory and electrical labour cost

The direct supervisory and electrical labour cost (DSELC) was found to be 10-25% of the operating labour (OLC). Now taking direct supervisory and electrical labour cost (DSELC) to be 17% of the operating labour (OLC), then

DSELC = 17% OLC

DSELC = ₦52, 542.92

Cost of utilities

The cost of utilities (UC) was found to be 10-20% of the total production cost (TPC). Now taking the cost of utilities (UC) to be 13% of the total production cost (TPC), then

UC = 13% TPC

UC = ₦40, 179, 88

Maintenance cost

The maintenance cost (MC) was found to be 2-10% of the fixed capital investment (FCI). Now taking the maintenance cost (MC) to be 5% of the fixed capital investment (FCI), then

MC = 5% FCI

MC = ₦72, 900

Operating supplies cost

The operating supplies cost (OSC) was found to be 10-20% 0f the maintenance cost (MC). Now, taking the operating supplies cost (OSC) to be 17% of the maintenance cost (MC), then

OSC = 17% MC

OSC = ₦12, 393

Laboratory charges

The laboratory charges (LabC) was found to be 10-20% of the operating labour cost (OLC). Now, taking the laboratory charges to be 13% of the operating labour cost (OLC), then

LabC = 13% OLC

LabC = ₦6, 830. 58

Plant overhead cost

The plant overhead cost (OVC) was found to be 50-70% of the sum of operating labour cost (OLC), operating supplies (OSC) and the maintenance cost (MC). Now, taking plant overhead cost (OVC) is 53% of the sum of operating labour cost, operating supplies (OSC) and the maintenance cost (MC), then

OVC = 53% (OLC + OSC + MC)

OVC = ₦137, 835. 92

General expenses

Administrative cost

The administrative cost (AC) was found to be 10-60% of the operating labour cost. Now taking the administrative cost (AC) to be 15% of the operating labour cost (OLC), then

AC = 15% OLC

AC = ₦7, 881. 44

Distribution and selling price

The distribution and selling price (PSP) was found to be 2-3% of the total production cost (TPC). Now taking the distribution and selling price (PSP) to be 3% of the total production cost (TPC), then

DSP = 3% TPC

DSP = ₦9, 272.28

Research and development cost

The research and development cost (RDC) was found to be 3% of the total production e production (TPC), then

RDC = 3% TPC

RDC = ₦9, 272.28

So, the general expenses (GE) are given as the sum of the administrative cost, distribution cost and selling price and research and development cost. That is,

GE = AC + DSP + RDC

GE = ₦26, 426

Total production cost

The total production cost (TPC) is given as the sum of the maintenance cost and the general expenses. That is

TprodC = MC + GE

TprodC = ₦99, 326

Further, the total expenditure, which is given as the sum of the raw material, factory labour, depreciation and overhead, is equal to

TE = RMC + OLC + Dep + OVC

TE = ₦490, 716, 84

Revenue expectation

On the basis that the plant is working for 156 days per year, that is, in this case, annum = 156 day the cost price is calculated as thus

Annum 156 day

V = 100 L/day

Amount per Annum = $156 \times 10^4 \times$ L/annum

Annum × V = 156×10^4 L

Price of Diesel ₦80/L

Having known the cost price, the selling price can be set, setting the selling price to be ₦80/L, that is

Profit = Amount per annum × price of Diesel.

Profit 1.248×10^4

Profit = profit. Annum

TI = profit

Gross income (GI) is the difference between the total income and the total expenditure (TE)

That is

GI = TI – TE

GI = ₦657, 957.16

The total income above is the income tax. Given that is 30% of the total income, that is

Tax = 30% GI

Tax = 197, 387. 108

Net profit (NP) is thus obtained as the difference between the gross incomes (GI) and the tax.

(Tax). That is,

NP = GI – Tax

NP = 460, 570. 012

Rate of return (RR) is given as

$$\text{Rate_of_return} = \frac{\text{Net_profit}}{\text{Total_capital_investment}} \cdot 100$$

So, symbolically,

$$\text{ROR} := \frac{\text{NP}}{\text{TCI}} \cdot 100 \cdot \%$$

ROR = 27.5%.

Cash flow

Cash flow is given as the difference between the amount earned and the amount expended. That is

Cash flow = TI - TprodC

Cash flow = ₦1, 148, 674

Pay back period

The pay back period (PBP) is the required period after the start of the plant to pay off the initial investment from income. It is given as the reciprocal of the rate of return (ROR)

That is

$$\text{PBP} := \frac{1}{\text{ROR}} \cdot \text{yr}$$

PBP = 4 yrs.

Discounted cash flow (Time value of money)

The discounted cash flow is used in plant analysis to obtain the net present value of the cash flow in year n. The net cash flow in each year of the plant is brought to its

present worth at the start of the plant by discounting it at some chosen compound interest rate.

The formula for the net present worth (NPW) of cash flow in year n is given as

$$NPW = \sum_{i=1}^{n} \frac{Cash_Flow}{(1+r)^n}$$

Now, for this plant,

$r := ROR$

$n := 1$

Therefore,

$$NPW := \sum_{i=1}^{n} \frac{Cash_Flow}{(1+r)^n}$$

NPW = ₦900, 920. 78

Discounted cash flow rate of return

The discounted cash flow is given as is the interest rate that will make the condition given as

$$DCF = \sum_{i=1}^{n} \frac{Cash_Flow}{(1+r)^n} = 0$$

Using trial-and-error calculations as suggested by Udeh and Keth (2001).

R =31.4%

$n := 20$

$DCFRR := r$

$$DCF := \sum_{i=1}^{n} \frac{Cash_Flow}{(1+r)^n}$$

$$DCF = 7.034 \times 10^3 \text{ Naira}$$

That is to say that the Discounted Cash Flow Rate of Return (DCFRR) is equal to

. DCFRR = 31.4%

Economic viability

The economic viability of this project can be concluded by testing with some principles laid down by Udeh and Keth (2001). The principles are:

i. If a project has a positive net present value (NPV) at the rate of return per annum calculated, then it is acceptable from the financial point of view. That means, the project is economic viable Udeh and Keth (2001).

ii. If the Discounted Cash Flow Rate of Return (DCFRR) of a project exceeds the target discount rate (that is, Rate of Return), the project is accepted, and otherwise, it is rejected Udeh and Keth (2001).

iii. Now, with regard to this design project, the Net Present worth (NPW) is ***positive*** and has a value of ₦900, 920. 78

. Therefore, based on the first principle of Udeh and Keth, the project is economic viable.

Further, the economic analysis reveals that the value of Discounted Cash Flow Rate of Return (DCFRR) which is 31.4% is greater than the value of Rate Of Return (ROR) which is 27.5%, this is another reason, based on the second principle of Udeh, that shows that the design project is economic viable.

Apart from that, another criterion used to judge the viability of a project is the Pay Back Period (PBP). The Pay Back Period of this project has been calculated to be approximately equal to 4 years. In essence, it means that the plant will pay back the initial investment in, at most, 4 years. This also shows that the project is economically viable provided cheap oil (non-edible) could be sourced.