

**STUDY ON PRODUCTION OF BIODIESEL FROM SELECTED
OIL BEARING NUTS AND UTILIZATION IN ENGINE**

*Thesis submitted in part fulfillment of the requirements for the award
of the degree of **Doctor of Philosophy in (Bioenergy)** to the
Tamil Nadu agricultural University, Coimbatore-03*

By

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DEPARTMENT OF BIOENERGY
AGRICULTURAL ENGINEERING COLLEGE
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TAMIL NADU AGRICULTURAL UNIVERSITY
COIMBATORE 641 003
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ABSTRACT
STUDY ON PRODUCTION OF BIODIESEL FROM SELECTED OIL
BEARING NUTS AND UTILIZATION IN ENGINE

By

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2003

Import of petroleum products constitutes a major drain on our foreign exchange reserves (Rs.90, 000 crores / annum). The acid rain, global warming and health hazards are ill effects of increased polluted gases like SO_x, CO and particulate matter in the atmosphere due to use of fossil fuels. Among the various alternative sources to petroleum products, oil from tree seed/crops have a potential for meeting the increasing requirements of diesel. Studies on the fuel properties of tree seed oils revealed that the kinematic viscosity of pungam, palm, pinnay, jatropha, neem, coconut and eluppai oils at 40°C was 8, 11, 8, 7, 7, 5 and 9 times higher than the recommended kinematic viscosity of diesel fuel respectively. The specific gravity was in the range of 0.91 to 0.92, flash point, cloud point and pour point were 211 to 288°C, 13 to 18°C and 1 to 13°C, carbon residue and ash

content were ranged from 0.45 to 0.64 per cent and 0.011 to 0.021 per cent respectively. The calorific values for tree seed oils were ranged from 38.28 to 40.17 MJ/kg. It is observed that the fuel properties of tree seed oils did not meet out the Indian standards for diesel fuel. These problems in diesel engine can be removed by using biodiesel obtained through transesterification of vegetable oils, which is ecofriendly. The biodiesel from seed oils has now become more economical as a diesel substitute. Hence an attempt was made to produce biodiesel from selected tree seed oils such as jatropha, pungam, coconut, palm, pinnay, neem and eluppai, and evaluate performance of the diesel engine with selected biodiesel.

The major obstacle in using the tree seed oils in engine was higher viscosity. By heating the oils above 100°C, the viscosity was brought to that of diesel fuel, whereby mere blending the oil with diesel, the maximum reduction in viscosity was achieved with 20 per cent raw oil blended with diesel. It had specific gravity equal to that of diesel, but 1 to 1.7 times higher viscosity than diesel. The above oils were found to be unsuitable for diesel engine.

During the production of biodiesel by the process of transesterification, methyl ester conversion rate of oils was ranged from 97.98 (neem oil) to 99.47 per cent (jatropha oil). Oils of eluppai and pinnay were failed to produce biodiesel and gel formation was observed due to higher free fatty acid content. The calorific values for produced biodiesels were ranged from 38.11 to 39.31 MJ/kg. The kinematic viscosity of biodiesel from pungam, palm, jatropha, neem and coconut oils at 40°C

was found to be 1/7.5, 1/9.3, 1/6.8, 1/6.5 and 1/5.5th of the viscosity of raw oil respectively. The specific gravity was ranged from 0.861 to 0.885, flash point, cloud point and pour point were 156 to 179°C, 12 to 16°C and -1 to 11°C, and carbon residue and ash content of biodiesels ranged from 0.24 to 0.32 per cent and 0.010 to 0.015 per cent respectively. The optimal conditions for best yield of biodiesel from pungam and jatropha oil were observed with catalyst amount of 0.3 and 0.2 M respectively, molar ratio of 1:6 and reaction time of two hours.

A biodiesel pilot plant used for jatropha biodiesel production, from 15 kg of jatropha oil, 14.9 kg of pure biodiesel and 2.25kg of glycerol was obtained. In jatropha biodiesel blended fuels, increase in amount of diesel fuel showed no effect on specific gravity of fuels. The viscosities of biodiesel-blended fuel have little effect on reducing the viscosity of biodiesel. The calorific value of blended fuels was improved by addition of diesel fuel in the biodiesel blends. The flash point of blended fuels was increased with increase in amount of biodiesel in the fuels.

The electrical generator coupled with a 5.2 kW diesel engine was used to test the performance and emission of biodiesel and its blends. In the case of jatropha biodiesel, the fuel consumption was about 14 per cent higher than that of diesel. The per cent increase in specific fuel consumption ranged from 3 to 14 for B₂₀ to B₁₀₀ fuels. The brake thermal efficiency for biodiesel and its blends was found to be slightly higher than that of diesel fuel at tested load conditions and there was no difference between the biodiesel and its blended fuels efficiencies.

The exhaust gas temperature increased with increase in load and consumption of biodiesel of jatropha and its blended fuels. The carbon dioxide emission by the use of biodiesel slightly increased than with diesel. The carbon monoxide reduction by biodiesel was 16, 14 and 14 per cent at 2, 2.5 and 3.5 kW load conditions. The nitrogen oxides emissions from biodiesel was increased by 15, 18 and 19 per cent higher than the diesel fuel at 2, 2.5 and 3.5 kW load conditions. At all tested load conditions, the oxygen emission level was almost same for jatropha biodiesel blended fuel and biodiesel when compared with diesel fuel.

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(D.RAMESH)

CERTIFICATE

This is to certify that the thesis entitled “**STUDY ON PRODUCTION OF BIODIESEL FROM SELECTED OIL BEARING NUTS AND UTILIZATION IN ENGINE**” submitted in part fulfillment of the requirements for the degree of **DOCTOR OF PHILOSOPHY IN BIOENERGY** to the Tamil Nadu Agricultural University, Coimbatore is a record of bonafide research work carried out by **Er. D. RAMESH** under my supervision and guidance and that no part of this thesis has been submitted for the award of any other degree, diploma, fellowship or other similar title or prizes and that the work has not been published in part or full in any scientific journal or magazine.

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ABBREVIATIONS USED

°C	-	Degree centigrade
kg	-	Kilogram
g	-	gram
mg	-	milligram
ppm	-	parts per million
lit	-	litre
ml	-	millilitre
h	-	hour
min	-	minute
secs	-	seconds
MJ	-	Mega Joule
μl	-	microlitre
w/w	-	weight by weight
KOH	-	Potassium hydroxide
NaOH	-	Sodium hydroxide
M	-	Molar weight of catalyst
N	-	Normality
Rs	-	Rupees
m	-	Metre
cm	-	Centimetre
mm	-	Millimetre
ha	-	Hectare

FAME	-	Fatty Acid Methyl Ester
IS	-	Indian Standards
cSt	-	centi Stokes
rpm	-	revolutions per minute
hp	-	horse power
BHP	-	Brake Horse Power
AC	-	Alternating Current
kW	-	kilo Watts
W	-	Watts
NO _x	-	Nitric oxide compounds
SO _x		Sulphur oxide compounds
NA	-	Not Applicable

CHAPTER I

INTRODUCTION

Oil is the major source (about 40%) of the world's primary energy consumption. India is currently the world's eighth largest consumer of oil and is expected to rise to fifth place in the next 20 years. The consumption and demand for the petroleum products are increasing every year due to increase in population, standard of living and urbanisation. The petroleum products continue to be the backbone of Indian economy with a share of 33 per cent of the energy basket. In India, there is a deficit of 40 per cent in supply of petroleum products and our petroleum reserves are limited to 6 to 7 years only (Elangovan, 2002). The increase in crude oil import affects the country's economy and its development. In India, the expenditure on crude oil imports has increased sharply from Rs.6,118 crores in 1991 to Rs.90,000 crores in 2002, which is 14.7 times higher than what it was before. This is also expected to rise continuously.

In India, the major diesel consuming sectors are transport, agriculture and industry. Diesel plays an important role in agricultural production for the operation of tractors, farm equipments, pump sets etc. Diesel consumption pattern in India has not varied much and is around 36×10^6 tonnes as reported by the Ministry of Petroleum and Natural Gas (MoPNG, 2003). The consumption of high-speed diesel oil (HSDO) during 1994-95 was 32.3×10^6 tonnes and it was 36.5×10^6 tonnes during 2001-02.

Apart from increase in fuel demand and limited petroleum reserves, diesel and other petroleum products are always under threat of supply instabilities and rise in oil prices in developing countries like India. Diesel engines are the major source of air pollutants released into the environment (Gassman, 1987). The exhaust gases contain oxides of nitrogen (NO_x), oxides of sulphur (SO_x), carbon monoxide (CO), unburnt hydrocarbon (HC) and particulate matter consisting primarily of soot. The diesel vehicles were banned in New Delhi and Bangalore cities for serious problem of air pollution due to higher emissions of polluted gases. The acid rain, global warming and health hazards are the results of ill effects of increased polluted gases like SO_x, CO and particulate matter in atmosphere. The aforementioned issues really worries the planners and policy makers and there is urgent need to search for suitable alternate diesel fuel from renewable sources.

When looking for alternate energy sources, attention is naturally focused on biomass as energy resource, which can make a significant contribution to satisfy the energy needs of society (Ruiz-Altisent, 1994). Among the various renewable energy sources, seed oil-bearing crops have a potential as supplement for meeting the increasing requirements of petroleum and its products. Vegetable oils from rapeseed, soybean, pongamia and jatropha seeds are some of the alternatives to diesel fuel. Raw vegetable oils from different oil crops were tried for running diesel engines by many researchers (Goering *et al.*, 1982; Korus *et al.*, 1982; Peterson *et al.*, 1983; Bagby, 1987; Juneja and Singh, 1995 and Kalam and Masjuki, 2002). But, they encountered problems like higher viscosity, poor volatility and poor flow characteristics

of vegetable oils, which caused severe engine deposits, injector coking and piston sticking in diesel engines (Peterson *et al.*, 1983). Acrolein is highly toxic substance emitted from the engine during thermal decomposition of glycerol present in vegetable oils (Schwab *et al.*, 1987). These problems can be eliminated by transesterification of vegetable oils to form biodiesel (Perkins *et al.*, 1991) and also the fuel properties of biodiesel can be made closer to the diesel fuel.

Transesterification technology provides biodiesel as a renewable fuel and glycerol as a byproduct, which can be a raw material for different chemical products. Biodiesel is produced by transesterification of large branched triglycerides of seed oil into smaller straight chain molecules of methyl esters (biodiesel), in presence of the catalyst. There are three stepwise reactions with intermediate formation of diglycerides and monoglycerides resulting in the production of three mol of biodiesel and one mol of glycerol. The biodiesel is biodegradable, non-toxic, has low emission profiles and it is environmentally beneficial (Krawczyk, 1996). Biodiesel has very low sulphur content and releases lower CO during combustion in diesel engines. Biodiesel contains oxygen, which leads to complete combustion in internal combustion (IC) engines. Biodiesel can be used in the existing diesel engines without any modification and can mix readily with diesel at any blending level (Krawczyk, 1996, and Srivastava and Prasad, 2000).

According to a survey of 28 countries by the International Energy Agency, biodiesel is reported to have been produced within the last six years in 21 countries, mainly in the European Union, East Europe, Malaysia and USA with an overall

capacity of around 13,00,000 tonnes. Indian Oil Corporation (IOC) developed multipurpose biodiesel pilot plant with capacity of 60 kg per day and Indian Railways and IOC are working together to develop biodiesel for captive use (Raje and Tuli, 2002).

In most of the developed countries, biodiesel is produced from soybean, rapeseed, sunflower, peanut, etc., which are essentially edible in the Indian context. Among the various vegetable oil sources, tree seed oils abundantly available in nature and they were selected for this study. Biodiesel source is selected based on oil content, availability and oil seed yield of the trees.

The tropical climate of India is conducive for several species capable of giving oil-bearing seeds. India has about 86 types of oil seed bearing perennial trees (Bringi, 1987). The oil plant, *Jatropha curcas* (L) is a multipurpose drought resistant large shrub or small tree. The biodiesel from jatropha oil has now become more economical as a diesel substitute. The first 1000 hectares of jatropha plantations were planted in Nicaragua, which yielded 1,600 tons of oil (Foidl *et al.*, 1996).

This jatropha oil covered 0.3 per cent of the diesel requirement of Nicaragua. Indian Railways conducted the first successful trial run of a super fast passenger train (Shatabdi Express from Delhi to Amritsar) on December 31, 2002 with five per cent of biodiesel blended with diesel as fuel.

The Government of Tamil Nadu is undertaking major venture of developing wasteland (3 lakh ha) in the state. The wastelands available in our country is 30 million hectares (Anon, 2002) in which Tamil Nadu state's share is 3 lakh ha. Realizing the importance of development of wastelands, the Government of Tamil Nadu has launched a massive wasteland development programme. Biodiesel crops have lot of scope to be planted in this land to meet out the increasing diesel demand apart from helping the farming community by creating rural employment and reducing the oil imports and air pollution.

Even though biodiesel production and uses has been reported by numerous authors (Abdul and Van Gerpen, 2001), each case reported to specific source of oil tested under experimental conditions. The effect of reaction of chemicals with the oils, their reaction and duration of purification have to be studied and standardised to suit the oils produced from native genotypes of the oil seed bearing trees (Vadivel, 2003).

Having analyzed the scope and importance of biodiesel from oil bearing tree seeds, the present study was taken up with the following major objectives.

- i. Screening, selection of tree seed oils and study the oil properties
- ii. Process development for biodiesel production from tree seed oils
- iii. Evaluate and compare the properties of the biodiesel with diesel fuel
- iv. Development of pilot biodiesel plant
- v. Performance evaluation of diesel engine with selected biodiesel fuel
- vi. Study the cost economics for biodiesel production.

CHAPTER II

REVIEW OF LITERATURE

Biodiesel is mono alkyl monoester made from vegetable oils by transesterification process. The oil can be obtained from soybean, canola, rapeseed, jatropha etc, or waste oil, animal fats etc. This renewable source is reported to be as efficient as petroleum diesel in powering unmodified diesel engine. This chapter deals with vegetable oil sources, characteristics of oils, methods of production of biodiesel and on their use in running the engine. This chapter divided into the following subtopics.

- i. Vegetable oil sources
- ii. Vegetable oils as engine fuel
- iii. Biodiesel production by transesterification of vegetable oil
- iv. Engine testing with biodiesel and biodiesel standards
- v. Cost economics

2.1. Vegetable oil sources

Goering *et al.* (1982) and Pryor *et al.* (1982) studied about more than 350 oil-bearing crops, among which only sunflower, safflower, soybean, cottonseed, rapeseed, jatropha seed and peanut oils were considered as potential alternative fuels for diesel engines.

Ramanathan (2001) reported seed oil yield from trees like *Macauba*, *tucuma*, *pique* and *jatropha* were ranged from 1.5 to 7.0 tonnes oil per ha per year. Hedge (2002) reported that oil production from the tree origin oil seeds in India

was estimated as 0.15 million tonnes. Eromosele and Paschal (2003) characterised viscosity of seed oils from wild plants and found that at 30°C the viscosity was reduced by 70 per cent their values. Properties of different oils are presented in the table 2.1 (LAAF, 1999).

Table 2.1. Comparison of fuel properties of fats and oils

Oil or Fat	Cetane Number	Heat of combustion, kJ/kg	Viscosity at 38 °C, cSt	Cloud Point, °C	Flash Point, °C
Castor	-	39500	297	-	260
Corn	37.6	39500	34.9	-1.1	277
Cottonseed	41.8	39468	33.5	1.7	234
Crambe	44.6	40482	53.6	10.0	274
Linseed	34.6	39307	27.2	1.7	241
Peanut	41.8	39782	39.6	12.8	271
Rapeseed	37.6	39709	37.0	-3.9	246
Safflower	41.3	39519	31.3	18.3	260
High-oleic safflower	49.1	39516	41.2	-12.2	293
Sesame	40.2	39349	35.5	-3.9	260
Soybean	37.9	39623	32.6	-3.9	254
Sunflower	37.1	39575	37.1	7.2	274
No.2 Diesel	47.0	45343	2.7	-15.0	52

Srivastava and Prasad (2000) reported that the vegetable oils had higher viscosity due to larger molecular mass and chemical structure. They also found that the presence of bound oxygen in vegetable oils lowered their heating values by about 10 per cent.

2.1.1. Promising tree seeds

Bringi (1987) reported that oil from the *bassia latifolia* kernel was 50 per cent. The main components of fatty acids in oil were 24 per cent palmitic acid and 37 per cent oleic acid. He also reported that fruits yield from the neem tree was ranged from 37 to 55 kg. The oil content in neem kernel and seed was 45 and 40 to 45 per cent respectively.

Ramanathan (2001) reported that palm oil obtained from the fruit pulp was 83 per cent and 17 per cent oil was obtained from palm kernel oil. The main components of fatty acids in palm oil were 48 per cent palmitic acid and 40 per cent oleic acid. He also reported that oil palm was the highest oil-yielding crop (3 to 5 tonnes per ha per year) and life span was 3 to 25 years.

Singh (2002) reported that coconut cultivated in India was 1.78 million ha and oil yield per ha was 2260 kg.

Bhasabutra and sutiponpeibum (1982) estimated the chemical composition of *Jatropha curcas* oil and reported to contain 21 per cent saturated fatty acids and 79 per cent unsaturated fatty acids.

The oil content of jatropha seed reported by different authors various from 30 to 40 per cent in whole seed and up to 55 per cent in kernel (Bhasabutra and Sutiponpeibum, 1982; Kumar and Kumar, 1984; Raina, 1984; Lahane and Relwani, 1986; Banerji *et al.*, 1985; Vimal and Tyagi, 1988 and Kandapal and Mira, 1995).

Banerji *et al.* (1985) studied the properties of four species of jatropha oil for energy and reported that the energy value of *jatropha multifida* was found to be higher than other species, while oil content was medium.

Jones and Miller (1992) reported that jatropha is a fast growing plant and can achieve a height of three meters within three years under a variety of growing conditions. The number of trees per hectare at planting can range from 1,600 to 2,200. Seed production ranged from about 0.4 to over 12 tonnes per ha per annum, after five years of growth.

Kandapal and Mira (1995) listed different names of *Jatropha curcas*. *Jatropha curcas* is commonly known as physic nut, Chandrajyoti, Chandraprabha or Nepalm. They also mentioned that each fruit of *Jatropha curcas* bears 2 to 3 seeds and weight of each seed ranged between 600 and 700 mg.

Vadivel (2003) reported that recommended jatropha plant spacing was 15 to 25 × 15 to 25 cm and 2 × 1.5 to 3 × 3 m for hedgerows in one or two rows and plantations respectively. The jatropha plant does not thrive in wastelands without water and two or three irrigation per month is required. Seed production from propagated plant was expected with 3 to 4 years and seed production ranges from about 0.4 tonne initially to 10 tonnes per ha per year. Oil bearing seeds from trees and their oil contents are presented in the table 2.2.

2.2. Vegetable oils as an engine fuel

Goering *et al.* (1982) reported that the major problems associated with the use of pure vegetable oils as fuel in diesel engines was high viscosity. It was

revealed that the vegetable oils were extremely viscous, with viscosities ranging from 10 to 20 times greater than No.2 diesel fuel.

Mori (1983) investigated engine performance with crude *Jatropha curcas* oil. He observed that an engine using curcas oil only stopped after 2h and when using 50 per cent curcas oil blended with No.2 diesel it stopped after 22h due to fuel filter block in the engine.

Harwood (1984) reported that the problem, probable cause and potential solutions for using straight vegetable oil in diesel engine. It was suggested that gum in the oil has to be removed or the oil to be converted to ester for solving short and long term problems.

Pestes and Stanislao (1984) studied the piston ring deposits in vegetable oil fuelled engine. The study revealed that the increased viscosity, low volatility and poor cold flow properties of vegetable oils were responsible for in severe engine deposits, injector coking and piston ring sticking.

Performance study of engine with sunflower oil as fuel was carried out by Abdul (1985). Problems like filter and fuel line blockage and poor atomization were encountered in the engine due to higher viscosity. Bagby (1987) reported that the higher viscosity of vegetable oils lead to incomplete combustion due to poor fuel atomization and inefficient mixing with air.

Hemmerlein *et al.* (1991) reported on rapeseed oil as a fuel and concluded that the physical and chemical properties are very similar to those of diesel fuel and on a long-term basis it can be used in diesel engines. They also reported comparative test results of six different engines fueled with rapeseed oil and diesel fuel.

Srinivasa rao and Gopalakrishnan (1991) studied four vegetable oils and their methyl esters as a fuel for diesel engine. They found drop in efficiency at full load due to poor combustion characteristic of vegetable oils because of higher viscosity and poor volatility.

Machacon *et al.* (2001) reported that use of vegetable oil such as coconut oil increased the brake specific fuel consumption and decreased the break mean effective pressure by 16 per cent.

Silvio *et al.* (2002) observed that high viscosity of pure vegetable oils reduced the fuel atomization and increased fuel spray penetration. They found that higher spray penetration was partly responsible for high engine deposits and thickening of lubricating oil.

2.3. Biodiesel production by transesterification of vegetable oils

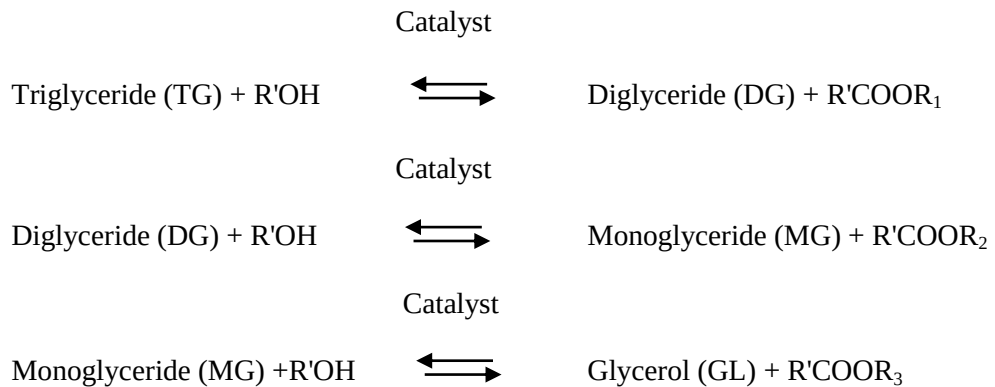
Since the vegetable oils are not ideally suited as fuels for a diesel engine, they have to be processed to bring their properties closer to diesel oil. This modification is mainly aimed at reducing the viscosity, which would eliminate

flow problems. The methods employed include (i) heating of the vegetable oil, (ii) thermal cracking of the highly complex vegetable oil molecule into lighter ones and (iii) transesterification, in which methanol/ethanol substitutes glycerol, splitting the vegetable molecule into lighter molecule (Srinivasa rao and Gopalakrishnan, 1991 and Srivastava and Prasad 2000).

Among these three methods, the transesterification of vegetable oil seems to be the best choice, as the physical characteristics of fatty acid esters (biodiesel) are brought close to those of diesel fuel (Schwab *et al.*, 1987).

2.3.1. Chemistry of transesterification process

General equation for a transesterification reaction is given below. The presence of a catalyst either a strong acid or catalyst base accelerates considerably the adjustment of the equilibrium.



Romano (1982) stated that residue of catalyst in the ester causes engine problems. He also found that base catalysts produced high levels of incombustible ash and acid catalysts attacked the engine metallic parts.

Transesterification process is used to remove the glycerol in triglycerides and replaces it with radicals from alcohol used for the conversion process (Kusy, 1982).

Freedman *et al.* (1984) explained various purification of ester obtained after the transesterification process.

Isigigur *et al.* (1994) suggested that chemical conversion of the oil by transesterification process with short chain alcohols, such as methanol or ethanol, to its corresponding fatty esters to be a most promising solution to the high viscosity problem. The transesterification process decreased the fuel viscosity and maintained the cetane number and heating values.

Ma *et al.* (1998) reported that the end products were mixture of esters, glycerol, alcohol, catalyst and tri-, di- and monoglycerides obtained after transesterification of triglycerides and that obtaining pure esters was not easy.

There are two types of transesterification process employed to produce biodiesel from vegetable oils viz; i. Catalyst transesterification (Ulf Schuchardt *et al.*, 1998 and Saka and Kusdiana, 2001) ii. Catalyst free transesterification.

A. Catalyst transesterification

The transesterification is a reversible reaction, in which excess alcohol is used to shift the equilibrium to the products side. Catalyst is usually used to improve the reaction rate of biodiesel production and biodiesel yield. Catalyst transesterification may be further classified into three types based on the nature of

catalytic used. They are alkaline catalyst based transesterification, acid catalyst based transesterification and enzyme catalyst based transesterification.

i. Alkaline Catalyst based transesterification

Tanaka *et al.* (1981) provided a novel method for preparation of lower alkyl, i.e. methyl esters of fatty acids by alcoholysis reaction of fatty acid glycerides of naturally occurring oils or fats, with a lower alcohol in a two-step process. An overall conversion of 98 per cent or more of the starting fatty glycerides was achieved.

Nye and Southwell (1983) studied production of biodiesel from rapeseed oil in a bench scale operation of 25 lit batches of oil in an enclosed stainless steel cylindrical drum with propeller driven by a 0.25 kW electrical motor. Rapeseed oil was added to a solution of one per cent KOH by oil weight, dissolved in six molar of methanol and stirred with splashing for one hour at 24°C. The mixture was allowed to settle to separate the phases of methyl ester and glycerine with unreacted methanol. The ester was washed and dried over anhydrous calcium chlorides and filtered.

A study on effects of different catalysts on methyl and ethyl esters of rapeseed oil at room temperature was conducted by Nye and Southwell (1984). They concluded that only alkoxides and hydroxides were effective at room temperature.

Wimmer (1992) blended 27.8 g of KOH, 240 g of methanol and 1618 kg of unrefined rapeseed oil and stirred the mixture for 20 min. With this 6.9 g of KOH

and 60 lit of methanol were added to the mixture. Then 80 kg of water was added and the mixture was allowed to stand overnight at room temperature. Glycerol was separated from the ester. The rapeseed oil methyl esters (less than 1.5 % remaining as glycerides and 0.008 % as ash) were used without further purification. The addition of water before removing glycerol formed emulsion, resulting in losses of esters and glycerol.

Zhang (1994) transesterified edible beef tallow with a free fatty acid content of 0.27 per cent. The tallow was heated to remove moisture under vacuum and kept at 60°C. Transesterification was conducted using one per cent NaOH (by the weight of tallow) dissolved in six mole of methanol at 60°C for about 30 min. After separation of glycerol, the ester layer was transesterified again using 0.2 per cent NaOH and 20 per cent methanol at 60°C for about one h. The purified ester was heated again to 70°C under vacuum to remove residual moisture. The laboratory scale process yielded 80 per cent ester.

Stern *et al.* (1995) patented a method to produce fatty acid esters from acid oil by transesterifying oil with glycerol to form glycerides. After transesterification, a large portion of glycerol was mixed with ester, wash water and neutralized with acid. The separated free fatty acids reacted with non-neutralized glycerol phase at about 200°C. The triglycerides (acidity of 3.2 per cent) from the reaction were added to the next alcoholysis step. The ester obtained from the "starting oil plus glyceride" had density of 880 kg/m³ and flash point of 185°C.

Foidl *et al.* (1996) developed a methodology for preparation of methyl ester and ethyl ester from *Jatropha curcas* L. oil. They used 0.53 mole of KOH and 10.34 mole of methanol for methyl ester production from jatropha oil at 30°C. They reported that the process duration was 11 hours and it was a two-step process. They also produced ethyl ester from jatropha oil by using 0.53 mole of KOH and 6.9 mole of ethanol at 75°C for 90 min.

Zamora *et al.* (1997) studied two-step transesterification process of *J. curcas* oil at laboratory level. Higher yield of biodiesel was obtained with 50 per cent excess of methanol of the stoichiometric quantity, 1.3 per cent of weight of KOH / NaOH (8: 2) and 75 per cent of the basic solution added at the first and 25 per cent at the second step.

Canakci and Van Gerpen (1999) observed that the best temperature was 60°C for methanol based transesterification process. They also suggested that the reaction temperature might be fixed at just below the boiling point of alcohol to provide favorable conditions for transesterification process.

The experimental conditions used by different authors for alkaline catalyst based biodiesel production from various vegetable oils are given in the table 2.3.

ii. Acid catalyst based transesterification

Sridharan and Mathai (1974) stated that the most preferable acid catalysts such as sulfuric, sulphonic and hydrochloric acid could be used in transesterification process to obtain higher yield of biodiesel. Nye *et al.* (1983) obtained 79.3 per cent ester

conversion with methanol using frying oil. They used H_2SO_4 as catalyst at 65°C for 40 h. Canakci and Van Gerpen (1999) observed 57.2 to 98.4 per cent conversion from soybean oil with different percentage of sulphuric acid over 48 h of reaction time.

iii. Comparison of performance of alkaline and acid catalysts in biodiesel production

Freedman *et al.* (1984) reported that alkali-catalyzed transesterification is much faster than acid-catalyzed transesterification. Comparative study on transesterification using acid and base catalysts (Schwab *et al.*, 1987 and Liu, 1994) resulted in acid catalyzed transesterification requiring heating to accelerate the reaction and that the time may vary from few min to several h.

Canakci and Van Gerpen (1999) concluded that the acid catalyst method did not gave high conversion rates but only under certain conditions with an ample reaction time.

Alcantara *et al.* (2000) reported that alkaline catalysts had higher rate of conversion from triglycerides to methyl esters especially if the acidity index is higher than 0.5 per cent. The comparative study indicated that the alkaline catalyst yielded 96 per cent ester conversion in about 3 h. Even after 40 h of processing of acid catalyst did not reach 20 per cent conversion for triglycerides having acidity index of higher than 0.5 per cent.

Saka and Kusdiana (2001) found that reaction time required for biodiesel production by using an alkaline catalyst was 1 to 8 h and 1 to 45 h for acidic catalysts.

iv. Enzyme based transesterification

Jackson and King (1996) reported a direct methanolysis of triglycerides using an immobilized lipase inflowing supercritical carbon dioxide. Corn oil was pumped in a carbon dioxide stream at a rate of four lit /min and methanol at five lit/min to yield more than 98 per cent fatty acid methyl esters. This process combined the extraction and transesterification of oil.

Kamini and Iefuji (2001) investigated methanolysis of vegetable oils in aqueous medium by *Cryptococcus* spp. S-2 lipase. They found that the optimal methanolysis conditions were oil/methanol molar ratio of 1:4 water content of 80 per cent by weight of the substrate containing 2000 U of crude lipase with shaking at 160 rpm for 120 h at 30°C and the reaction was conducted in a single step to avoid the stepwise addition of methanol. They reported that the methyl ester contents reached 80.2 per cent (by wt.) at 120 h.

B. Catalyst free transesterification

Saka and Kusdiana (2001) investigated transesterification reaction of rapeseed oil in supercritical methanol without using any catalyst. They carried out the experiment in a batch-type reaction vessel preheated at 350 and 400°C, at a pressure of 45 to 65 MPa and with a molar ratio of 1:42 of rapeseed oil to methanol. They found that preheating temperature of 350°C for 240 sec of supercritical treatment of methanol was sufficient to convert the rapeseed oil to methyl esters.

Ayhan Demirbas (2002) studied biodiesel production from vegetable oils by transesterification using supercritical methanol. He reported that the methyl ester of vegetable oil or biodiesel is very similar to diesel fuel. Its viscosity is only twice that of diesel fuel and its molecular weight was roughly 1/3 that of vegetable oil.

2.3.2. Factors affecting biodiesel yield in transesterification process

The biodiesel yield is affected by different parameters such as moisture and free fatty acids of oil, molar ratio of oil and alcohol, catalyst type and amount, reaction time, reaction temperature and stirring speeds.

i. Moisture and free fatty acids

Ma *et al.* (1998) studied the effects of free fatty acids and water on transesterification of beef tallow with methanol. They reported that the water content of beef tallow should be kept below 0.06 per cent w/w and free fatty acid content of beef tallow should be kept below 0.5 per cent (w/w) in order to get the best conversion. Water content was a more critical variable in the transesterification process than free fatty acids.

ii. Molar ratio

Tanaka *et al.* (1981) developed two-step transesterification of oils and fats for tallow, coconut oil and palm oil by using 6:1 to 30:1 molar ratios with alkali catalyst and methyl ester. The yield of esters was found to be of 99.5 per cent.

Nye and Southwell (1983) investigated the methanolysis of rapeseed oil by using one per cent NaOH or KOH catalyst. They found that the molar ratio of 6:1 of methanol to oil gave the best conversion.

Freedman *et al.* (1984) studied the effect of molar ratio (from 1:1 to 6:1) on ester conversion with different vegetable oils. The study showed that soybean, sunflower, peanut and cottonseed oils behaved similarly and achieved highest conversions (93 to 98 per cent) at a 6:1 molar ratio. Molar ratios greater than 6:1 did not increase the yields but complicated the recovery of FAME and glycerol and increased the cost of alcohol recovery.

Freedman *et al.* (1986) found that one of the most important variables affecting the yield of ester was molar ratio of alcohol to triglyceride. The molar ratio was associated with the type of catalyst used. An acid catalyzed reaction needed 30:1 ratio of BuOH to soybean oil while an alkali-catalyzed reaction required only a 6:1 ratio to achieve the same ester yield for a given reaction time.

A study conducted by Zhang (1994) recorded that molar ratio of 6:1 was optimum for beef tallow transesterification with methanol. In the laboratory study, 80 per cent by tallow weight of esters was recovered.

Mohmed *et al.* (2002) found out that 100 per cent excess ethanol reduced the reaction time significantly.

iii. Catalyst type

Freedman *et al.* (1984) chose sodium hydroxide to catalyze in transesterification process. They found that the ester conversions at the 6:1 ratio for one per cent NaOH and 0.5 per cent NaOCH₃ were almost the same after 60 min.

They also studied the transesterification of soybean oil with methanol, ethanol and butanol using one per cent concentrated sulfuric acid. It was shown that a 30:1 ratio resulted in a high conversion to methyl ester and it was unsatisfactory when the molar ratios were 6:1 and 20:1.

Liu (1994) reported that sodium methoxide was more effective because of the assumption that a small amount of water was produced upon mixing NaOH and MeOH.

iv. Reaction time

Freedman *et al.* (1984) conducted experiments for transesterification of peanut, cottonseed, sunflower and soybean oils under the condition of methanol to oil ratio of 6:1, 0.5 per cent sodium methoxide catalyst and 60°C. They found that yield of 80 per cent was observed after one min for soybean and sunflower oils and also observed that after one h, the conversions were almost same for all four oils (93 to 98%).

Ma *et al.* (1998) studied the effect of reaction time on transesterification of beef tallow with methanol. The reaction was very slow during the first min due to the mixing and dispersion of methanol into beef tallow. From one to five min the reaction proceeded very fast. The production of beef tallow slowed down and reached the maximum value at about 15 min. The di- and monoglycerides increased at the beginning and then decreased. At the end the amount of monoglycerides was higher than that of diglycerides.

v. Reaction temperature

Freedman *et al.* (1984) studied the transesterification of refined soybean oil with methanol (6:1) using one per cent NaOH for three different reaction temperatures. They reported ester yields of 94, 87 and 64 per cent for 60, 45 and 32°C respectively after 0.1 h of the transesterification process. They observed the ester formation was identical for the 60 and 45°C runs and only slightly lower for the 32°C run after one h. They concluded that temperature clearly influenced in the reaction rate and yield of esters.

vi. Stirring speed

Peterson *et al.* (1992) studied the effect of agitation on the transesterification of vegetable oil and observed that higher agitation promoted the homogenization of the reactants.

Ma *et al.* (1999) studied the effect of mixing on transesterification of beef tallow. Mixing was very important to the transesterification of beef tallow because melted beef tallow and sodium hydroxide methanol solution (NaOH-MeOH) were immiscible. They found that there was no reaction without mixing. NaOH-MeOH was added to the melted beef tallow without stirring and with higher stirring speeds. Longer stirring times were needed to mix the two phases subsequently and in both cases, the reaction was started after mixing of two phases and stirring was no longer needed.

Alcantara *et al.* (2000) narrated importance of stirring speeds for the reactant mixtures to yield methyl esters. It was observed that stirring at 360 rpm for two h had less than ten percent yield while 600 rpm for two h of stirring reached over ninety percent yields. Stirring of mixture was required to speed up the reaction in alkaline catalyst transesterification process.

2.4. Engine testing with biodiesel

Mittelbach *et al.* (1985) conducted experiments for emissions from two different rapeseed methyl esters in engine. These two biodiesel gave significantly lower total particulates and lower polymer aromatic hydrocarbons than No. 2 diesel fuel. They also found that combustion of methyl ester fuels produced higher levels of NO_x emissions and aldehyde emissions than No.2 diesel.

Ouedraogo *et al.* (1991) experimented with rapeseed and jatropha oils and their ethyl esters as fuel in a 2.8 kW four cycle diesel engine. They observed a 15 per cent power drop with the esters. Maximum torque produced using the oil and esters were less than that diesel. They reported that the specific fuel consumption of esters was greater than diesel fuel.

Srinivasa rao and Gopalakrishnan (1991) studied four vegetable oils and their methyl esters as a fuel for diesel engine. The study indicated that thermal efficiency and maximum power output of methyl esters of vegetable oils were close to the diesel values.

Krahl *et al.* (1992) reported that emissions of hydrocarbons, CO and NO_x were lower with rapeseed methyl ester as compared to diesel.

Fosseen and Goetz (1993) carried out experiment to reduce NO_x emission using a 20/80 methyl soyate / No.2 diesel fuel by shifting diesel injection timing. They observed a significant reduction in NO_x with a shift in beginning of injection from 0 to -3° while the reduction of other emissions was retained.

Marshall (1993) studied the effects of methyl esters of tallow and grease on exhaust emissions in a Cummins L10 engine with different level of esters in fuel blends varying from 0 to 100 per cent. He reported that reductions in CO and particulate emissions ranged from 15 to 50 per cent and 0.11 per cent increase in NO_x per percentage of tallow ester in the fuel. Formaldehyde and aldehyde emissions were not significantly different to those for the diesel fuel. Fuel consumption was increased because of reduced heating value of the ester fuels.

Schumacher *et al.* (1993) investigated the emission and performance of tractor engine with soydiesel and soydiesel/diesel blends. They observed an average power difference of less than 0.4 per cent in tractor fuelled with a 20 per cent soydiesel and 80 per cent of No.2 diesel as compared to No.2 diesel only. High reduction in power was observed when tractor engines were fuelled with 100 per cent soydiesel.

Pandey (1996) reported that by using biodiesel as fuel in five hp direct injection engine the smoke number increased with load with higher rate of increased at higher loads.

Peterson *et al.* (1997) reported that the average thermal efficiencies for biodiesel fuel in the mapping test was insignificant by difference when compared to No.2 diesel. The average fuel consumption on mass basis was 7 per cent higher than diesel.

Schumacher (1999) studied soybean methyl ester as a fuel in a Dodge truck. He reported that it could be used in diesel engines with little or no difficulty. When using 10-20-30-40-50 per cent (volumetric) soybean methyl ester-diesel fuel mixtures, power, smoke intensity, CO and HC emissions decreased, NO_x emissions and fuel consumption increased.

Abdul and Van Gerpen (2001) reported that neat biodiesels and their blends were similar to No.2 diesel fuel with the same thermal efficiency but higher fuel consumption.

Lue *et al.* (2001) carried out experiments on production of biodiesel from waste fried vegetable oil and investigated emission characteristics of a small Direct Injection (D.I) diesel engine using biodiesel blends and diesel fuels. They used 20/80 and 30/70 blends of biodiesel to diesel fuel in this study. They found that diesel engine fuelled with biodiesel blends emits more PM₂ particle number concentrations than those with diesel fuel and PM₂ number concentration increased as biodiesel concentration increased. Smoke and gaseous emissions such as CO, HC, NO_x and SO₂ results favored biodiesel blends.

Wu *et al.* (2001) reported emission characteristics of a small D.I diesel engine using biodiesel-blended fuels. They reported that biodiesel produces less carbon monoxide, hydrocarbons, nitrogen oxides and sulfur dioxides and slightly increases in the amount of particulate matter released.

Cardone *et al.* (2002) carried out tests in a turbocharged direct injection passenger car diesel engine fuelled with 100 per cent *Brassica carinata* biodiesel and compared with diesel fuel. They found that the engine test bench analysis did not show any appreciable variation of output engine torque values when compared with diesel fuel and significant difference in specific fuel consumption data at the lowest loads for the biofuels and diesel fuel. They also conducted engine heat release analysis and result showed that there was a potential for increased thermal NO_x generation when firing biodiesel with no prior modification to the injection timing. They reported biodiesel emissions contained less soot as compared with diesel fuel and a greater fraction of the particulate was soluble.

Dorado *et al.* (2003) studied exhaust emissions from a diesel engine fuelled with transesterified waste olive oil. They reported that biodiesel resulted in lower emissions of CO (up to 58.9 %), CO₂ (up to 8.6 %), NO (up to 37.5 %), and SO₂ (up to 57.7 %), with increase in emissions of NO₂ (up to 81 %) as compared with diesel fuel. They also found that combustion efficiency remained constant using either biodiesel or diesel fuel and increase in brake-specific fuel consumption recorded in the case of biodiesel.

2.4.1. Standards for biodiesel

During transesterification process intermediate glycerols mono and diacylglycerols are formed and small amounts of which can remain in the final biodiesel (methyl ester) product. Besides these partial glycerols unreacted triacylglycerols, unseparated glycerol, free fatty acids, residual alcohol, and catalyst can contaminate the final product. The contaminants can lead to severe operational problems when using biodiesel, such as engine deposits, filter clogging or fuel deterioration (Knothe, 2001).

Kusy (1982) computed the conversion of methyl ester from triglycerides by the amount of glycerol present in methyl ester. Romano (1982) computed the methyl ester conversion rate on the basis of viscosity and density difference between oil and its methyl esters.

De Filippis *et al.* (1995) applied viscometry method for determining conversion of vegetable oil to methyl ester. They observed the viscosities determined at 20°C and 37.8°C were good agreement with GC analyses conducted for verification purposes.

Gelbard *et al.* (1995) studied yield of transesterification reaction by ¹H- nuclear magnetic resonance (¹H-NMR) and developed a simple equation for measuring methyl ester conversion rate.

$$C = 100 * (2A_{ME}/3A_{T-CH_2})$$

where

C - Conversion of vegetable oil to corresponding methyl ester

A_{ME} - Integration value of the protons of methyl esters

A_t-CH_2 - Integration value of the methylene protons.

In some European countries such as Austria, the Czech Republic, France, Germany and Italy standards have been developed to limit the amount of contaminants in biodiesel fuel. The biodiesel standards used in different countries and Indian requirement for diesel fuels are presented in the tables 2.4 and 2.5.

2.5. Cost economics for biodiesel production

Melville (1987) studied zero profit analysis of cost of methyl ester of winter rapeseed from two different sizes of units. Cost of production for biodiesel as reported in the table 2.6.

Table 2.6. Cost of production for biodiesel derived from winter rapeseed oils

Particulars	Biodiesel plant size	
	Co-op sized unit	Farm sized unit
Crushing capacity, t/year	1480	78.6
Production of methyl ester, lit /year	655000	34000
Production cost of the ester, \$/lit	0.31	0.425
By product costs		
Price of glycerol, \$/kg	0.93	0.93
Soybean meal, \$/ton	110.23	110.23

Caringal (1987) estimated the cost of production for methyl ester of winter rapeseed based on plant processing 10 metric tonnes of rapeseed oil per day. He found that the cost of production for methyl ester of winter rapeseed was

\$0.346/lit by considering rapeseed oil at \$0.44/kg, methanol at \$0.158/lit and KOH at \$ 1.00/kg.

Reed *et al.* (1995) estimated cost of biodiesel from new vegetable oil and waste vegetable oil. They reported that the processing costs were estimated as \$0.50/gallon above the feedstock cost. They concluded that cost of biodiesel from new vegetable oil and waste vegetable oil was \$ 2.5 and \$ 1.50/gallon, respectively.

Van Dyne *et al.* (1996) studied the microeconomic effects of a community based biodiesel production system. The value of products and co-products from the biodiesel plant are presented in table 2.7.

Table 2.7. Cost economics for biodiesel from soybean oil

Particulars	Units / bushel	Biodiesel production, \$/bushel
Biodiesel at \$0.84/gallon	0.82 gallons	0.70
Soybean meal at \$233/ton	0.27 tonnes	6.29
Glycerin at \$ 0.30/ #	0.6 pounds	0.18
Total products and co-products value	-	7.17
Farm gate price of soybeans	-	5.60
Net increase in value (\$/bushel)	-	1.57

A review of 12 economic feasibility studies showed that the projected costs for biodiesel from oilseed or animal fats ranged from US\$0.30 to 0.69/lit including the meal and glycerin credits and the assumption of reduced capital investment costs by having the crushing and/or esterification facility added onto an existing

grain or tallow facility. The cost of biodiesel from vegetable oil and waste grease reported as US\$ 0.54 to 0.62/lit and US\$ 0.34 to 0.42/lit, respectively (Bender 1999).

CHAPTER III

MATERIALS AND METHODS

This chapter deals with selection of raw materials for biodiesel production and methods adopted to study the properties of tree seed oils. Method of production of biodiesel and procedure followed in assessing the properties of biodiesel, performance and emission profile of biodiesel-fuelled diesel engine is also explained. This chapter is divided into following subtopics.

- i. Selection of oils for biodiesel production
- ii. Properties of selected tree seed oils
- iii. Production of biodiesel from selected tree seed oils
- iv. Properties of biodiesel
- v. Optimal conditions for biodiesel production
- vi. Biodiesel pilot plant
- vii. Performance of biodiesel fuelled engine
- viii. Cost economics.

3.1. Selection of oils for biodiesel production

The tree seed oils selected for biodiesel production are based on the local availability. Among various vegetable oil sources, seed oils from the following tree species were selected for this study and table 3.1 describes the oil content, oil seeds yield per ha and its potential.

Botanical name	Tamil name	Plant and seeds/nuts
<i>Pongamia glabra</i>	Pungam oil	Plate No. 3.1
<i>Elaeis guineensis</i> Jaq	Ennai panai oil	Plate No.3.2
<i>Calophyllum inophyllum</i>	Pinnay oil	Plate No.3.3
<i>Jatropha curcas</i> .L	Kattanamakku oil	Plate No.3.4
<i>Azadirachta indica</i>	Vembu oil	Plate No.3.5
<i>Cocos nucifera</i>	Thengai oil	Plate No.3.6
<i>Bassia latifolia</i>	Eluppai oil	

3.2. Properties of selected tree seed oils

Fuel and chemical properties of selected tree seed oils are the important parameters for the selection of alternatives to diesel fuel to use in diesel engines. The instruments used to test the fuel properties of oils are shown in plate 3.7. The following fuel properties, which are commonly studied for diesel fuels were selected for this study (IS 1460:2000 and Pramanik, 2002).

- i. Calorific value
- ii. Kinematic viscosity
- iii. Specific gravity
- iv. Flash point
- v. Cloud point
- vi. Pour point
- vii. Carbon residue
- viii. Ash content

The following chemical properties tree seed oils were selected for this study (Canacki and Van Gerpen, 1999 and Tomasevic and Siler-Marinkovic, 2003).

- i. Free fatty acid
- ii. Acid value
- iii. Total glycerol content
- iv. Free glycerol content
- v. Combined glycerol content

3.2.1. Methods adopted for properties measurement

The procedure followed to study the fuel and chemical properties of selected oils are detailed below.

A. Fuel properties

Studies on fuel properties of the selected tree seed oils helps in assessment of suitability as fuel in diesel engine. In addition to that, they give an idea about post engine operations such as emissions, power out put etc.

i. Calorific value

Most important property for alternate fuels is heat of combustion, which determines the power out put and thermal efficiency of engines. The calorific value of tree seed raw oils were determined by IS:1448-1960 method and compared with diesel fuel. An adiabatic oxygen bomb calorimeter was used for this study.

ii. Kinematic viscosity

The kinematic viscosity of samples was determined by Redwood viscometer using American Standard Testing Methods (ASTM) 445-72 method. The viscometer water bath was heated to few degrees above testing temperature. The test sample was filtered in B200 mesh and poured into oil cup of viscometer. The sample was stirred to make it homogeneous and the viscometer bath was adjusted to achieve testing temperature. A 50 ml measuring cylinder was put at few cm below the jet of viscometer. The time taken for 50 ml oil sample filling was noted after lifting the ball valve in oil cup of viscometer. The kinematic viscosity of sample was calculated by using the equations 3.1 and 3.2. Each experiment was replicated for three times.

$$\text{Kinematic viscosity of test sample, cSt} = 0.26 \times t - 179/t \quad \dots (3.1)$$

$$\text{for } 34 < t < 100$$

$$= 0.24 \times t - 179/t \quad \dots (3.2)$$

$$\text{for } t > 100$$

where, t – time taken for the fall of 50 ml of oil sample, secs

iii. Specific gravity

The specific gravity of tree seed oils was determined by specific gravity bottles method using IS : 1448-1972. The experiments were conducted at 40°C.

$$\text{Specific gravity of test sample} = \frac{W_3 - W_1}{W_2 - W_1} \quad \dots (3.3)$$

where,

w_1 - weight of empty specific gravity bottle

w_2 - weight of water + specific gravity bottle

w_3 - weight of test sample + specific gravity bottle

The same procedure was followed for blended fuels also.

iv. Flash point

The Pensky Martens closed flash point tester was used to determine the flash point of oils (IS:1448-1992 method). The test was performed by filling the oil cup up to a definite mark, fixing the cover and heating the oil at a rate of 5 to 6°C per min. A stirrer was used to mix and get uniform temperature distribution in the test sample. The flash point was recorded at the time of distinct flash in the interior of oil cup by reading temperature. The experiment was conducted three times for each test fuels and average value of flash point was reported.

v. Cloud point

The cloud and pour point apparatus was used to find out the cloud point of test samples. The ASTM method D-97/57 was adopted for this experiment. The test jar with oil was subjected to preliminary treatment. In this treatment, the oil was heated to 46°C and cooled to 15.5°C in water bath maintained at 7°C. After pretreatment, the ring jacket was placed at 25 mm from bottom of test jar and the test jar was kept in the jacket. The capillary thermometer was immersed 3 mm above the bottom of the test jar and the jacket was cooled between –1 to + 2°C.

The sample was frequently inspected at interval of 3°C by removing test jar from the jacket and observing crystal formation in the oil due to cooled bath in the apparatus and that temperature was recorded as cloud point of test sample. Each inspection was completed within five secs.

vi. Pour point

The pour point of test sample was determined by the same procedure followed for cloud point except for the position of the thermometer. The thermometer was immersed to 3 mm below the surface of the test sample. Inspection was continued until there was no movement of oil at horizontal position of test jar for five secs. The temperature recorded under these conditions was taken as the pour point of the test sample.

vii. Carbon residue

The carbon residue apparatus was used to determine the carbon residue of the oils. The experimental procedure was as per in ASTM D524-IP14/65 method. Ten g of test sample was weighed in a porcelain crucible and the porcelain crucible was placed in Skidmore crucible. Skidmore crucible was kept in an iron crucible and was loosely covered. The iron crucible was subjected to high strong flame by gas burner. The pre ignition period was 10 ± 0.5 min. Then the gas burner was tilted after the appearance of smoke and the flame intensity was reduced when the ignited vapours burn uniformly. The burning time for the vapour was 13 ± 1 min. The iron crucible was heated to cherry red hot and was

maintained for 7 min after observing no smoke emits from the apparatus. Total period used for heating was 30 ± 2 min. At the end of heating period, test crucible containing carbon residue was cooled in a desiccator and weighed. The carbon residue was calculated as percentage of the original sample and reported as Conradson Carbon Residue (CCR).

$$\text{Carbon residue in 10 g sample} = (\text{carbon residue} + \text{porcelain crucible weight}) - (\text{empty porcelain crucible weight}) \quad \dots (3.4)$$

$$\text{Carbon Residue (CCR), \%} = \text{Carbon residue in 10 g sample} \times 10$$

viii. Ash content

Ash content of test fuels was analyzed by IS:1448-1992 method. Hundred g of test fuel was taken in 120 ml silica crucible. The sample was heated between 700 and 800°C to obtain carboneous residue and the residue was heated in muffle furnace at $775 \pm 25^\circ\text{C}$. The experiment was continued until the consecutive weighing did not differ by more than 0.5 mg. The crucible was cooled in a desiccator and weighed. The ash content was expressed in per cent on mass basis using the following formula.

$$\text{Ash, percent by mass} = 100m/M \quad \dots (3.5)$$

where,

m – Mass of ash, g

M – Mass of sample, g

B. Chemical properties

The chemical properties of raw oil give an idea about the possibility of biodiesel production and its effect on biodiesel yield. The following chemical properties were studied for the selected tree seed oils.

i. Free fatty acid

The Association of Official Chemists Society (A.O.C.S) Official Method Ca 5a-40 was used to determine the Free Fatty Acid (FFA) of oils. Oil sample of 7.05 ± 0.5 g was weighed in conical flask. Seventy five ml of ethanol and 2 ml of phenolphthalein were added to the sample. The oil sample was titrated against 0.25N sodium hydroxide, shaking the flask vigorously until the appearance of first permanent pink color and the colour must persist for 30 secs.

The percentage of FFA content was calculated as oleic acid in most of fats and oils except coconut and palm oil as given below:

$$\text{FFA as oleic, (\%)} = \frac{\text{ml of alkali consumed} \times N \times 28.2}{\text{Weight of sample, g}} \quad \dots (3.6)$$

$$\text{For coconut, (FFA as lauric, \%)} = \frac{\text{ml of alkali consumed} \times N \times 20}{\text{Weight of sample, g}} \quad \dots (3.7)$$

$$\text{For palm oil, (FFA as palmitic, \%)} = \frac{\text{ml of alkali consumed} \times N \times 25.6}{\text{Weight of sample, g}} \quad \dots (3.8)$$

where, N - Normality of sodium hydroxide solution

ii. Acid value

The acid value is defined as the number of milligrams of KOH necessary to neutralize one gram of sample. The acid value of test sample was calculated by using A.O.C.S Official Method Ca 5a-40.

$$\text{Acid value} = \text{Free Fatty Acid of the sample} \times 1.99 \quad \dots (3.9)$$

iii. Total glycerol content

A.O.C.S Ca14-56 method was used to determine the total glycerol content of test sample. Sample size of 2 g was taken in an Erlenmeyer flask and 50 ml of alcoholic potassium hydroxide solution was added. The air condenser was connected to the flask and the sample was gently boiled for 30 min. Ninety nine ml of chloroform was added to one lit volumetric flask and then 25 ml of glacial acetic acid was added. The saponification flask was removed from the water bath and the sample was transferred to volumetric flask using 500 ml of distilled water. The stoppered flask was shaken vigorously for 30 to 60 secs. Distilled water was added up to mark and mixed thoroughly by inverting it and was set aside until aqueous and chloroform layers separated. Fifty ml of aqueous layer was added to the beaker containing 50 ml of periodic acid reagent. Two blanks were prepared by adding 50 ml of distilled water to beaker containing 50 ml of periodic acid reagent. Beakers were covered with watch glass and allowed for 30 min. Before titrating the samples, 20 ml of potassium iodide solution was added, mixed and allowed to stand

for one min. The sample was diluted with distilled water and resultant compound was titrated against the sodium thiosulphate solution. The titration was continued until brown iodine color almost disappeared. Two ml starch indicator was added to solution and the titration continued until blue iodine – starch complex colour just disappeared. The total glycerol content was calculated by

$$\text{Total glycerol content, \%} = \frac{(B-S) \times N \times 2.302}{W} \quad \dots (3.10)$$

where,

S-Volume of titrant, ml of sample

B- Volume of titrant, ml of blank

N- Normality of sodium thiosulphate solution used

W-Weight of sample represented by aliquot (ml) taken for the sample analysis

$$W = \frac{\text{Mass of sample} \times \text{ml of sample}}{900} \quad \dots (3.11)$$

iv. Free glycerol content

Free glycerol content of tree seed oils was determined using A.O.C.S Ca15-46 method. Sample size of 10 g was taken in a weighing dish. Ninety ml of chloroform was used to wash the sample weighing dish into one litre volumetric flask.

Distilled water of 500 ml was added and the flask was vigorously shaken for 30 to 60 secs. The distilled water was added up to mark, stoppered and mixed thoroughly by inverting and setting aside until aqueous and chloroform layers separated. Hundred ml of aqueous layer was added to beaker containing 50 ml of periodic acid reagent. Two blanks were prepared by adding 100 ml distilled water to beaker containing 50 ml of periodic acid reagent. Beakers were covered with watch glass and allowed for 30 min. Before titrating the samples, 20 ml of potassium iodide solution was added, mixed and allowed to stand for one min. The sample was diluted with distilled water and the resultant compound was titrated against sodium thiosulphate solution, the titration was continued until brown iodine colour almost disappeared. Two ml starch indicator was added to solution and the titration was continued until blue iodine starch complex color just disappeared. The free glycerol content was calculated by using the equation 3.12.

$$\text{Free glycerol content, \%} = \frac{(B-S) \times N \times 2.302}{W} \quad \dots (3.12)$$

where,

S-Volume of titrant, ml of sample

B- Volume of titrant, ml of blank

N- Normality of sodium thiosulphate solution used

W-Weight of sample represented by aliquot (ml) taken for the sample analysis

$$W = \frac{\text{Mass of sample} \times \text{ml of sample}}{900} \quad \dots (3.13)$$

v. Combined glycerol content

The combined glycerol content of the sample was calculated by the difference in total glycerol content and free glycerol content in the test sample and expressed in percentage.

3.3. Production of biodiesel from selected tree seed oils

The oils are composition of three moles of fatty acids and one mole of glycerol (triglycerides). The problem with substituting the oils for diesel fuels is mostly associated with their high viscosities, low volatilities and polyunsaturated character (Srivastava and Prasad, 2000). These can be modified by one of the following methods (Schwab *et al.*, 1987; Yusulf Ali and Hanna, 1994 and Srivastava and Prasad, 2000).

- i. Heating the raw oils
- ii. Blending of raw oil with diesel fuel
- iii. Transesterification
- iv. Thermal cracking and
- v. Microemulsification.

The last two methods require expensive equipment to convert the vegetable oil into biofuels. In the thermal cracking, high temperature (200 to 850°C) is involved in the conversion process. Emulsification can be achieved by mixing of alcohol with oils and yielded low energy content and cetane number fuels (Ma *et al.*, 1999). So the first three methods were considered for the present study and transesterification method was selected and compared with first two methods.

A. Heating the raw oils

The experiment was conducted by heating the raw tree seed oils to 100°C and kinematic viscosity was measured at an interval of 10°C reduction in temperature. The kinematic viscosity of selected tree seed oils at testing temperature was measured by Redwood viscometer. The effect of temperature on kinematic viscosity was studied.

B. Blending of raw oils with diesel fuel

The selected tree seed oils were investigated for their suitability for engine running and their effect on blending with diesel. The blending ratios used for selected oils with diesel are presented in table 3.2. The selected tree seed oils were blended with diesel at different ratios and experiments were conducted to measure kinematic viscosity of blended fuels as described in section 3.2.1. The influence of blending on kinematic viscosity of oils was studied and compared with diesel.

Table 3.2. Blending of oils with diesel fuel

Sl. No.	Notation used for blending proportion	Amount of blending, %	
		Oil	Diesel
1	B ₀	0	100
2	B ₂₀	20	80
3	B ₄₀	40	60
4	B ₆₀	60	40
5	B ₈₀	80	20
6	B ₁₀₀	100	0

C. Transesterification

In the transesterification of tree seed oils, triglyceride of oil reacts with alcohol added in the presence of a strong acid or base, producing a mixture of fatty acids alkyl ester and glycerol. The fatty acid mono alkyl ester obtained from the transesterification reaction is called as biodiesel. The stoichiometric reaction requires one mole of a triglyceride and three moles of the alcohol. Excess of the alcohol is used to increase the yields of the alkyl esters and to allow its phase separation from the glycerol formed. A laboratory scale biodiesel reactor was developed and used for biodiesel production from the selected oils. The process development for biodiesel production from tree seed oil is shown in Fig. 3.1.

i. Laboratory scale biodiesel reactor

A laboratory scale biodiesel reactor of one lit capacity was developed to produce biodiesel from selected tree seed oils (Fig. 3.2). The reactor consists of

three-neck flask of glass, water-cooled condenser, thermometer, mechanical stirrer and water bath. The condenser was used to condense methanol vapour releasing from the reactor and return the condensed methanol to reactor. Thermometer was used to measure the reactants mixture temperature during the experiment and vigorous mixing of reactants was done by mechanical stirrer.

Among various transesterification methods like alkaline, acid and enzyme catalyst based transesterification, alkaline-based transesterification method was selected for the study. Since the alkaline catalyzed method was 4000 times faster than that same amount of acidic catalyst (Formo, 1954). Because of faster reaction rate, better biodiesel yield, lesser duration, this method is most often used commercially than other mentioned methods above (Hideki Fukuda *et al.*, 2001).

Sodium hydroxide was used as catalyst in the transesterification reaction as this is cheaper than other alkaline catalysts. According to Canacki (2001) the catalyst amount used to convert the high FFA oils into biodiesel was 0.1 to 1.2 per cent in two steps. Most of the researchers used the amount 0.1 to 1.2 per cent for biodiesel production from low FFA oils up to 0.5%w/w (Freedman *et al.*, 1984; Ma *et al.*, 1999 and Alcantara *et al.*, 2000). In this study the catalyst amount was fixed at minimum of 1.2 per cent by weight basis of oil and was increased based on free fatty acid of tree seed oils.

For preparation of sodium methoxide solution NaOH pellets were completely dissolved in the methanol. The NaOH pellets were reacting with

water and CO₂ present in the atmosphere. During the reaction the NaOH reacted with CO₂ and yield sodium carbonate. This sodium carbonate affects the performance of the catalyst during the transesterification reaction. So after weighing of the NaOH pellets it was dissolved in methanol without delay.

The reaction temperature should always be below the boiling point (65°C) of methanol (Freedman *et al.*, 1984 and Canacki and Van Gerpen 1999) and the reaction temperature was fixed at 60°C for biodiesel production.

The reaction time for alkaline transesterification reaction varied between 1 to 8 h (Saka and Kusdiana, 2001). So for this study the reaction was stopped after 3 h and the methyl ester conversion of the biodiesel was determined.

The ratio of alcohol to tree seed oil is one of important parameter, which influences the transesterification reaction. According to Ulf Schuchardt *et al.* (1998), excess of alcohol favours the formation of the products and also that lead to recovery of the glycerol difficult. So the ideal alcohol / tree seed oil ratio was established empirically to the each individual process. Methanol was used for biodiesel production from selected tree seed oils. The reasons for selecting methanol were the low molar weight, low cost and lesser amount of alcohol required. For the calculation of molecular weight of the oil, the fatty acid composition of the oil was identified.

ii. Fatty acid methyl ester composition

The samples were prepared by diluting ester in hexane and used in gas chromatograph equipped with flame ionization detector. The column used in gas

chromatograph was a stainless steel column packed with 15 Carbowax-20 per cent M (coated on chromosorb G). The Gas Chromatograph (GC) and High Performance Liquid Chromatograph (HPLC) setup is shown in the plate 3.8.

The analytical conditions used in Gas Chromatograph (FID)

Oven temperature : 230 °C
 Injector temperature : 270 °C
 Detector temperature : 270 °C
 Carrier gas : Nitrogen - flow rate @30ml/min
 Zero air - flow rate @30ml/min
 Fuel gas : Hydrogen - flow rate @300ml/min

iii. Experimental conditions for the selected tree seed oils

The experimental conditions used for production of biodiesel from the selected tree seed oils in the laboratory scale reactor were described in table 3.3.

Table 3.3. Experimental conditions used for biodiesel production

Sl. No.	Oil used	Molar ratio	Reaction time, h	Catalyst amount*, %
1	Pungam	1:9	3	1.2
2	Palm	1:9	3	1.2
3	Pinnay	1:9	3	1.2
4	Jatropha	1:9	3	1.2
5	Neem	1:9	3	1.2
6	Coconut	1:9	3	1.2

7	Eluppai	1:9	3	1.2
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*** Catalyst amount increased for higher FFA content of the oils.**

The reactor was kept in water bath at 60°C. Oil sample of 500 g was used in this laboratory study. Freedman *et al.*, (1984) reported that ninety eight per cent of methyl ester conversion was achieved at molar ratio of 1:6, but even higher molar ratio up to 45:1 was necessary when oil contained larger amount of FFA. So the molar ratio of oil to methanol ratio used in the present study was 1:9 of methanol to oil ratio. Initially a reaction time of 3 h was used for transesterification of oils then the reactants were poured into separating funnel and kept for 12 h to separate the glycerol. Glycerol settled at the bottom and the crude biodiesel above the glycerol layer was collected. The collected biodiesel was washed three times with water and the glycerol content in biodiesel was determined by A.O.C.S Ca14-56 method as described in section 3.2.1. The methyl ester conversion was calculated based on glycerol content in the crude oil and biodiesel.

Procedure followed for production of biodiesel from selected tree seed oils

- i. The Free Fatty Acid (FFA) of oil was determined by A.O.C.S method.**
- ii. Based on the FFA content the catalyst amount was fixed.**
- iii. The molar ratio of oil: methanol used for the biodiesel production from the selected tree seed oils was kept 1:9.**
- iv. The tree seed oil of 500 g was poured into the laboratory biodiesel reactor.**
- v. Water-cooled condenser and thermometer with cork were connected to laboratory reactor.**
- vi. The catalyst NaOH was weighed and dissolved completely in methanol by using mechanical stirrer.**
- vii. The NaOH was dissolved in methanol to form sodium methoxide solution.**
- viii. The sodium methoxide was added to oil for vigorous mixing by mechanical stirrer.**
- ix. The reaction temperature (60°C) was maintained for 3 h and after completion of reaction time the reactant mixture was poured into separating funnel.**
- x. The glycerol was separated by gravity settling and removed from the separating funnel.**
- xi. The raw biodiesel remains suspended at the top of glycerol layer.**

xii. The raw biodiesel collected and by water washing the pH of biodiesel was brought down to seven.

xiii. This pure biodiesel was used for determining the methyl ester conversion rate. The methyl ester conversion was determined by A.O.C.S Ca 14-56 method. Canaki and Van Gerpen (1999) used this method to measure the methyl ester conversion rate of soya biodiesel production by acid catalyst based transesterification reaction. The procedure for methyl ester conversion rate estimation was given in section 3.4.C.v.

iv. Methods adopted for biodiesel production from higher FFA oil

The hypothesis of the study was higher FFA content ($>7\%$) of the oils neutralized by adding excess amount of catalyst. Ma *et al.* (1998) suggested that more NaOH can be used to neutralize the free fatty acids (FFA) if the acid value was greater than one. In the present study the same catalyst in different concentrations was used to create good conditions for better methyl ester conversion in the biodiesel production. The selected reaction time and catalyst amount for this study were 2 h and 0.3 M respectively. Three methods were adopted to produce the biodiesel from oils from these oils. The experiments were replicated three times and average values were presented. The details of the experimental study are given below.

a. One step method

The catalyst was dissolved in the methanol to form sodium methoxide solution. This solution was mixed with higher FFA content of oil during the transesterification reaction.

b. Two step method

The experimental design for biodiesel production with different combinations of concentrated sodium methoxide solution used in this study is presented in the table 3.4. For all reactions a part of sodium methoxide solution was added to oil after reaching the reaction temperature and a second part was added at the end of 0.5 h of transesterification reaction. The sodium methoxide solution prepared for I and II methods is shown in the table 3.4.

Table 3.4. Experimental design for biodiesel production from higher FFA oils

Two step method	Catalyst amount, Mole	Combinations of concentrated sodium methoxide solution used (1 st part: 2 nd part)		
I method	0.3	50:50	75:25	25:75
II method	0.3 (3/4:1/4)	50:50	75:25	25:75

c. Third method

Oil with high FFA content was made to undergo the pretreatment for conversion of FFA into esters. In this treatment the oil was mixed with sulphuric acid (1% by weight basis) and stirred for 1 h to make homogenous solution of the reactants. After the treatment the oil was kept for 8 h or over night. This second reaction was between oil and sodium methoxide. The molar ratio used for this

method was 1:30 and reaction times used in temperature acid treatment and alkaline treatments were 1 and 2 h respectively.

3.4. Properties of biodiesel

The properties of biodiesel were determined for assessing the fuel quality and comparing the biodiesel produced with international biodiesel standards. During the transesterification process intermediate glycerols mono- and diacylglycerols were formed small amounts of which can remain in the final biodiesel (methyl ester) product. Besides these partial glycerols unreacted triacylglycerols, unseparated glycerol, free fatty acids, residual alcohol, and catalyst can contaminate the final product. The contaminants can lead to severe operational problems when used in engine such as engine deposits, filter clogging, or fuel deterioration. Obtaining pure ester was not easy since there were impurities in the ester such as di - and monoglycerides, etc. The monoglycerides caused turbidity (crystals) in the mixture of esters (Ma, *et al.*, 1998). In some European countries, such as, Austria, Czech Republic, France, Germany and Italy standards have been developed that limit the amount of contaminants in biodiesel fuel. In these standards restrictions are placed on the individual contaminants by inclusion of items such as free and total glycerol for limiting glycerol and acylglycerols, flash point for limiting residual alcohol, acid value for limiting free fatty acids and ash value for limiting residual catalyst (Knothe, 2001).

A. Ultimate analysis

The ultimate analysis gives the elemental carbon, hydrogen, oxygen, sulphur and nitrogen of the sample. The ultimate analysis of biodiesels was carried out using a Carlo Ebra Elemental Analyzer (EA 1108) (Plate 3.9) coupled with auto sampler (AS-200) and Data Processor (DP-200-PRC) following the procedure suggested by ASTM D3174-76 standards. Analytical conditions maintained for CHNS determination were:

Oxidation furnace temperature	=	1200°C
Reduction furnace temperature	=	650°C
Oven temperature	=	60°C
Filament temperature	=	190°C
Carrier gas	=	Helium
Measuring flow rate	=	100 ml min ⁻¹ at 0.85 kpa
Reference flow rate	=	40 ml min ⁻¹ at 1.20 kpa
Oxygen flow rate	=	20 ml min ⁻¹ at 1.0 kpa
Sample delay time	=	11 secs
Sample delay stop	=	40 secs
Oxygen injection stop	=	50 secs
Peak enable stop	=	20 secs
Total run time	=	650 secs
Sample preparation	=	Oven dried
Sample weight	=	Approximately 3 mg

B. Fuel properties

The procedures for determining the fuel properties of biodiesels were same as used for the raw oils.

C. Chemical properties

The procedure used in the study of chemical properties of biodiesel was same for properties like acid value, free glycerol content and total glycerol content as followed for the oil. The following chemical properties of biodiesel were studied.

- i. Free fatty acid
- ii. Total glycerol content
- iii. pH
- iv. Sodium content
- v. Methyl ester conversion

i. Free Fatty Acids

The A.O.C.S Official Method Ca5a-40 was used to determine the free fatty acid of biodiesels. The oil sample of 28.2 ± 0.2 g was weighed in conical flask. Fifty ml of ethanol and two ml of phenolphthalein was added to the sample. The oil sample was titrated against 0.1N sodium hydroxide, shaking vigorously until the appearance of first permanent pink color and the color must persist for 30 secs and the FFA was calculated as detailed in section 3.2.1.

ii. Total glycerol content

A.O.C.S Ca15-46 method was used to determine the total glycerol content of biodiesels. The biodiesel sample, alcoholic potassium hydroxide and chloroform used for this experiment were 10 g, 100 ml and 91 ml respectively.

iii. pH

The pH value shows the presence of catalyst in the biodiesel. The pH was determined for crude and washed biodiesels. pH meter was used to determine the pH of biodiesel sample.

iv. Sodium content

The sodium content of crude and washed biodiesel was tested and compared. The sodium content was determined by using flame photometer. Before biodiesel sample was fed into the meter, meter was standardized. Standardization was done by feeding 10 to 1000 ppm sodium concentrated samples in to the flame photometer. The readings were plotted against sodium concentrations for standard curve. The test sample was fed in to the instrument and readings were plotted. Concentrations of both biodiesel samples were obtained from standard curve.

v. Methyl ester conversion

The complete of transesterification reaction was determined by A.O.C.S Official Method Ca 14-56 is known as Total, Free and Combined glycerol method. This method was used to find out the glycerol content in biodiesel and raw oils. Based on the glycerol content in the biodiesel and raw oils, the methyl ester conversion was calculated. The methyl ester conversion was calculated by using the following formula,

$$\text{Methyl ester conversion, \%} = \frac{(\text{Glycerol content in oil} - \text{Glycerol content in biodiesel})}{\text{Glycerol content in raw oil}} \times 100 \dots (3.14)$$

3.5. Optimal conditions for biodiesel production

Based on the FFA content, five tree seed oils were taken for study of biodiesel production. The Completely Randomized Design (CRD) was used to predict optimal conditions for biodiesel production from selected seed oils. The treatment design was 2×2 factorial arrangement. For this study the reaction temperature was fixed at 60°C and the reaction time was reduced from 3 to 2 h and the methyl ester conversion of the biodiesel was determined. There were two factors; catalyst amount and molar ratio as described in transesterification reaction conditions. The sample size taken for this study was 200 g. The experiment was replicated three times and average value of methyl ester conversion reported.

a. Catalyst amount

Three levels of catalyst amount viz. 0.1, 0.2 and 0.3 M (by molar weight basis) were used in the biodiesel production. Ma *et al.* (1999) and Alcantara *et al.* (2000)

reported that catalyst amount 0.1 to 1.2 per cent was used for biodiesel production from low FFA oils up to 0.5%w/w.

b. Molar ratio

Oil:methanol ratios of 1:4.5, 1:6 and 1:7.5 (50, 100 and 150 % excess of methanol of the stoichiometric quantity) were used to study their effect on biodiesel yield.

The molecular weight of the oil was calculated based on fatty acid compositions of the oil.

Five tree seed oils tested for biodiesel production, two oils viz., pongamia and jatropha were selected for further study to find the optimal conditions for higher yield of biodiesel. The parameters considered to find out higher biodiesel production were catalyst amount, molar ratio and reaction time. The effective reaction time for better biodiesel yield from these oils was identified. A Completely Randomized Design (CRD) was used and three levels of catalyst amount and molar ratios and four levels of reaction time on biodiesel yield were investigated. The treatment design was 3×3 factorial arrangements. There were two factors, reaction time and molar ratio as described in transesterification reaction conditions. The sample size used in this study was 200 g and experiment conducted twice and average value of methyl ester conversion reported. In this study, the molar ratio up to 1:6 was used and its effect on biodiesel yield was studied.

i. Analysis of raw oil and biodiesel in HPLC

a. Sampling

After transesterification reaction, the sample poured into separating funnel and kept for 12 h to separate the glycerol. The water used to wash the crude biodiesel sample and the pH was brought to 7.0. The moisture present in the sample was removed by passing through calcium carbonate. A sample of washed biodiesel was dissolved in 2-propanol-hexane (5:4) to yield the 1 per cent stock solution used to produce calibration solutions for UV detection at 205nm. Same procedure was adopted to prepare the stock solution for raw oil. The injection volumes of 20 μ l and flow rate of 0.5 ml/min were used for all experiments.

b. HPLC parameters

The response in mv-sec of HPLC to the fatty acid compositions of oil and methyl ester were obtained from Shimadzu model LC-6A with "winacds" software package. The following operating parameters were followed for the determination of compositions in samples.

Mobile phase	: Methanol (A) : 2-propanol-hexane (5:4 v/v) (B), (50%A+50%B)
Column	: C ₁₈ -pack CLC-ODS (M)
Flow rate	: 0.5 ml/min
Wave length	: 205 nm
Retention time	: ~ 15 min

c. Comparison

The retention time of the components of oil sample was compared with biodiesel and commercial diesel for a qualitative assessment as the standards for the individual components could not be imported.

3.6. Biodiesel pilot plant

The biodiesel pilot plant was developed at Department of Bioenergy and the capacity of developed biodiesel pilot plant was 250 lit of biodiesel production per day. The process flowchart for biodiesel and by product production from tree seed oils was shown in the Fig. 3.3 and the process development for biodiesel pilot plant was shown in the Fig. 3.4. The pilot plant consists of biodiesel reactor with heating and agitating device, catalyst-mixing tank, glycerol settling tanks and biodiesel washing tank. This pilot plant was developed for biodiesel production from different tree seed / vegetable oils.

3.7. Performance of biodiesel fuelled engine

The performance of engine was tested with selected biodiesel and its blends. A vertical four stroke, single cylinder Kirloskar diesel engine was used in the experiments without any modifications. The diesel engine coupled with AC alternator was used to conduct the engine testing with *Jatropha curcas* biodiesel and its blends. The engine testing setup consists of engine coupled with alternator, fin type air heaters for electrical load. A hole was made in the exhaust pipe to take the sample for analysis of the exhaust gases from the engine by mean of an exhaust

analyzer. The four levels of jatropha biodiesel blending at 20, 40, 60 and 80 per cent (B₂₀, B₄₀, B₆₀ and B₈₀) with diesel were used for engine testing.

The technical specification of diesel engine is given below.

Manufacturer	:	Kirloskar engines Ltd, Pune, India
Capacity	:	5.2 kW
BHP of engine	:	7
Speed	:	1500 rpm
Mode of injection	:	DI
Type	:	AV1

The technical specification of AC alternator is as follows.

Manufacturer	:	Kirlosker Electric Co Ltd., Bangalore, India
Volts	:	240
Amphere	:	14.6
Speed	:	1500 rpm
Phase	:	single
Cycle	:	50 Hz
KVA	:	3.5
Pf	:	1

The details of fine type air heater used for loading of the engine are furnished in table 3.5.

Table 3.5. Details of fins air heater

Sl. No.	Electrical heating heater capacity, W	Number of heaters used
1	2000	1
2	1000	1
3	500	2

A. Engine testing

The engine was tested with three electrical load levels viz., 2, 2.5 and 3.5 kW. The engine was started with diesel and changed to the desired blend. During engine testing the observations were recoded at one h interval during the engine testing. The following observations were made during the engine testing.

- i. Fuel consumption
- ii. Speed of engine crankshaft
- iii. Alternator output
 - a. Volts
 - b. Amphere
- iv. Power output of the engine
- v. Specific fuel consumption
- vi. Brake thermal efficiency

- iv. Emission profile
 - a. Excess air utilized
 - b. Exhaust gas and ambient temperature
 - c. Carbon dioxide (CO₂)
 - d. Carbon monoxide (CO)
 - e. Oxides of Nitrogen (NO_x)
 - f. Oxygen (O₂)

B. Methods used for measurement of parameters

The methods used for the measurement of above parameters are described as below.

i. Fuel Consumption (FC)

Fuel consumption was determined by allowing fuel from a graduated burette, noting down the time of consumption of 50 ml of fuel and calculating the hourly consumption.

ii. Engine crankshaft speed

The speed was measured using a hand held tachometer on the camshaft and doubling the value to get the speed of engine.

iii. Alternator output

Voltmeter and ammeter were used to measure the volts and current produced from the AC alternator. The electrical output of the AC alternator coupled

with diesel engine was measured at three different load conditions (2, 2.5, and 3.5 kW) for the selected biodiesel and its blends and compared with diesel.

iv. Power output of the engine (P)

The Power output of the engine was calculated from the electrical output of the alternator. The following formula was used to calculate the power in put from the engine to electrical generator.

$$\text{Power output of the engine, } P = \frac{\text{Volts} \times \text{Amperes}}{746 \times \text{efficiency of generator}} \quad \dots (3.15)$$

The efficiency of the generator was taken as 0.8.

v. Specific Fuel Consumption (SFC)

From data collected, the specific fuel consumption with the tested fuels was calculated as per I.S.I procedures (I.S.I., 1980).

$$\text{SFC} = \frac{(\text{mf}/\text{T}) \times 3600}{\text{P}} \quad \dots (3.16)$$

where,

- SFC - Specific fuel consumption, g/kWh
- mf - Mass of the fuel consumed, g
- T - Time period for the fuel consumptions, s
- P - Power out put of the engine, kW.

vi. Brake Thermal Efficiency (BTE)

The brake thermal efficiency was computed by using the following equation ((I.S.I., 1980).

$$\text{BTE} = \frac{3600 \times 100}{\text{SFC} \times \text{GHV}} \quad \dots (3.17)$$

where,

- BTE - Brake thermal efficiency, per cent
- GHV - Gross heating value, MJ/kg

vii. Exhaust profile

A KM900 exhaust gas analyzer was used to determine the composition of exhaust gases. The exhaust gas analyzer had sensors for NO_x, SO_x and CO to detect the exhaust gas composition. The ambient and exhaust gas temperatures

were also recorded by using KM900 exhaust gas analyzer attached with a k type thermocouple. The excess air, flue gas and ambient temperatures, CO, CO₂, NO_x and O₂ were recorded by using this analyzer and all data were stored. The duration used to test the engine with selected biodiesel and its blends was 2 h and the readings were measured at a time interval of 30 min.

3.8. Cost economics

The straight-line method was used to calculate the cost economics for biodiesel production using fixed and variable costs.

The following formulae were used for this study

$$\text{Depreciation} = \frac{\text{Initial value} - \text{salvage value}}{\text{Life period}} \quad \dots (3.18)$$

$$\text{Benefit Cost ratio (BCR)} = \frac{\text{Expenditure incurred}}{\text{Income}} \quad \dots (3.19)$$

The table 3.6 shows the electrical energy consumed by different components in the biodiesel pilot plant.

Table 3.6. The electrical consumption per batch of biodiesel production

Sl.No	Components	Capacity, W	Run time per Batch, h	Electrical energy used, Wh
1	Steam generator	200	2.00	400.0
2	Oil lift pump (2 No.s)	250	0.15	37.5
3	Mechanical stirrer	500	2.00	1000.0
4	Air compressor	200	3.00	600.0

Assumptions used in the cost economics calculation

Capital cost of the plant	=	Rs. 150000/-
Salvage value of the pilot plant	=	10 per cent of the capital cost
The interest on capital investment	=	12 per cent
Housing cost per year (Rent)	=	Rs.25000/-
Electrical charges	=	Rs. 4/- per kWh
Labour charges per day per person	=	Rs. 120/-
Repair cost per annum	=	Rs. 7500/- (5% of capital cost)

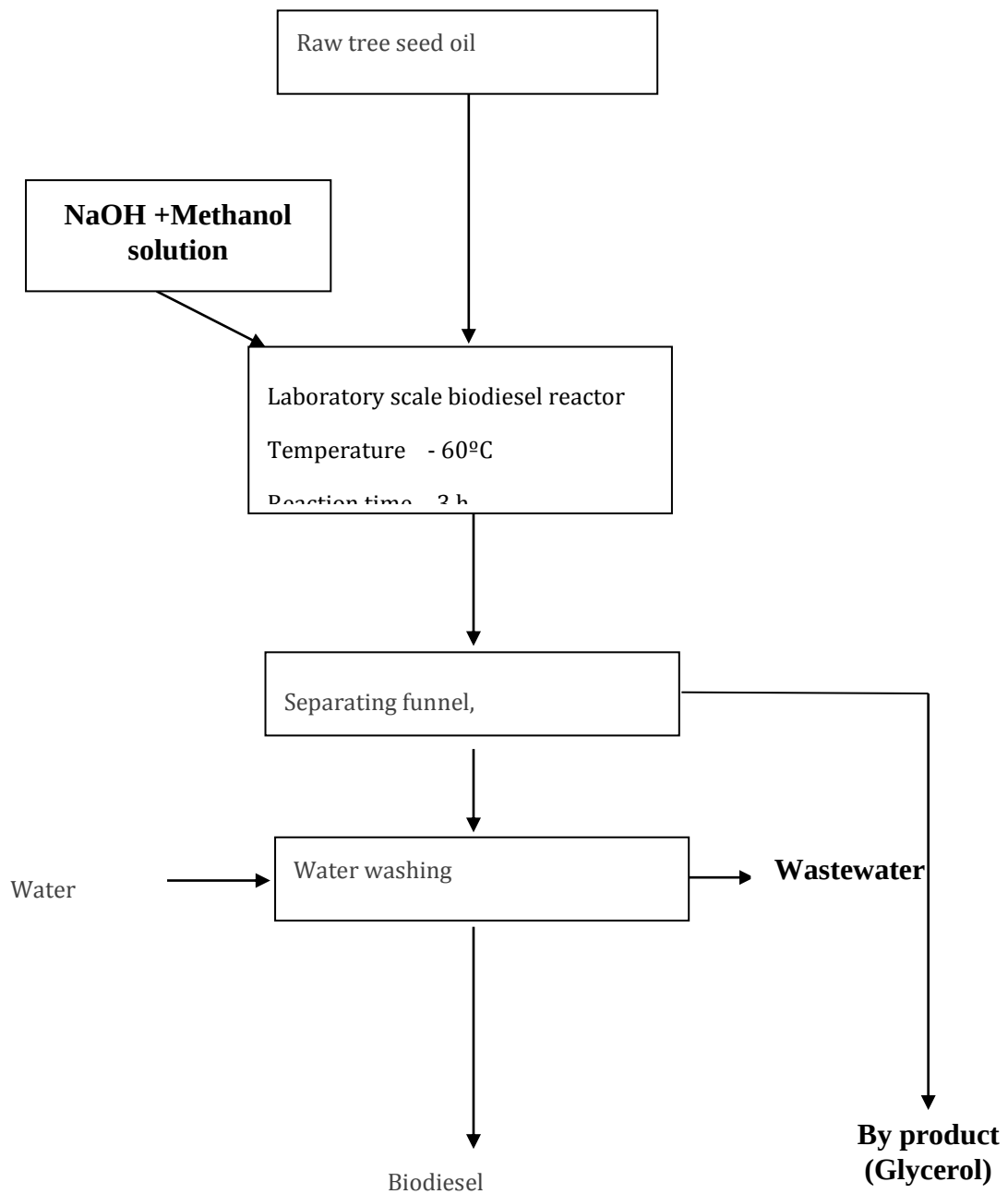


Fig. 3.1. Process development for biodiesel production from tree seed oil

CHAPTER IV

RESULTS AND DISCUSSION

The results of the investigations carried out to study the properties of selected tree seed oils, biodiesel production, properties, performance and emissions of biodiesel-fuelled engine are presented and discussed in this chapter. This chapter is divided into the following headings.

- i. Selection of oils for biodiesel production
- ii. Properties of selected tree seed oils
- iii. Production of biodiesel from selected tree seed oils
- iv. Properties of biodiesel
- v. Optimal conditions for biodiesel production
- vi. Biodiesel pilot plant
- vii. Performance of biodiesel fuelled engine
- viii. Cost economics.

4.1. Selection of oils for biodiesel production

The oil content of selected tree seeds varied from 27 to 83 per cent of the seed. The objective of this study is to exploit the potential of tree born seed oil sources for biodiesel production. The selection of tree seed oils was based on maximum oil content, seed yield per tree and easy availability. Among various viable sources, seven tree species yielding high oil productivity per seed were selected for this study. The selected seven trees whose seed oils studied were

pungam oil (*Pongamia glabra*), palm oil (*Elaeis guineensis* Jaq), pinnay oil (*Calophyllum inophyllum*), jatropha oil (*Jatropha curcas*), neem oil (*Azadirachta indica*), coconut oil (*Cocos nucifera*), and eluppai (*Bassia latifolia*).

4.2. Properties of selected tree seed oils

The fuel and chemical properties were assessed as stated in section 3.2 of chapter III to find the suitability for diesel engine and are presented in table 4.1.

4.2.1. Fuel properties

The fuel properties of oils were determined to compare with the standards of conventional diesel fuel. The experimental procedures and instruments used for finding the properties of oils were given in section 3.2.1. The fuel properties of oils are discussed below.

i. Calorific value

The calorific value is an important parameter for alternate fuels which directly related to fuel consumption. The calorific value of diesel fuel was 44.42 MJ/kg. The calorific values for tested oils ranged from 38.28 to 40.17 MJ/kg. From the table 4.1 it was observed that the calorific values of oils such as pungam, eluppai, pinnay, palm and jatropha were in a closer range. The higher heating values of oils ranged from 86 to 90 per cent to that of diesel fuel. The decrease in heat content of oils may be due to the greater unsaturation of hydrogen in the molecule. The maximum value was observed for

eluppai oil (40.17 MJ/kg) and low value for neem oil (38.28 MJ/kg). Goering *et al.* (1982) and Silvio *et al.* (2002) reported similar results for vegetable oils.

ii. Kinematic viscosity

The kinematic viscosity normally ranged from 2 to 5 and 2.5 to 15.7 cSt at 40°C for high speed diesel (HSD) and light diesel oil (LSD) respectively (IS 1460: 2000). The fuel injection system in diesel engines is designed to operate with the above recommended kinematic viscosity of diesel fuel. Using red wood viscometer it was observed that the kinematic viscosity of diesel fuel at 40°C as 4.86 cSt.

Kinematic viscosity at 40°C for pungam oil was observed as 41.89 cSt, which is 8.6 times more when compared to diesel fuel viscosity. Srinivasan (2003) reported that kinematic viscosity of pungam oil was 74.14 cSt at 30°C and 10 times higher than the viscosity of diesel fuel which was due to fatty acid composition and oil test temperature.

The kinematic viscosity of palm oil was observed as 54.70 cSt and this is 11.3 times more than viscosity of diesel fuel at 40°C. This value was closer to the value reported by Bari *et al.*, (2002).

For pinnay oil the kinematic viscosity was 40.87 cSt. The kinematic viscosity of jatropha oil was 36.25 cSt which was 7.5 times higher than the recommended kinematic viscosity of diesel fuel. The kinematic viscosity of neem

oil was observed as 36.75 cSt. The lowest value of 27.48 cSt was observed for the coconut oil. Kinematic viscosity of eluppai oil was 45.32 cSt which was 9.3 times of that diesel fuel at same temperature.

In general, it was noted that the kinematic viscosity of raw oils was 6 to 11 times higher than desirable viscosity of diesel fuel at 40°C. From experimental data it was inferred that higher viscosity levels (27.48 to 54.70 cSt) of oils are not suitable for running diesel engines. Bhasabutra and Sutiponpeibun (1982); Srinivasa rao and Gopalakrishnan (1991); Foidl *et al.* (1996) and Srivastava and Prasad (2000) confirmed these results. This indicates that viscosity of tree seed oils did not meet the requirements of Indian standards for diesel fuel.

iii. Specific gravity

Specific gravity of the tested oils ranged from 0.9102 to 0.9261 (table 4.1), which was higher as compared with diesel fuel (0.8396). The specific gravity of jatropha oil was 0.9098 at 40°C. Bringi (1987) reported the same value. The specific gravity of coconut, pinnay and jatropha oils were found to be closer to the specific gravity of diesel fuel. The maximum value of specific gravity was observed for palm oil and minimum for jatropha oil.

The specific gravity is an important parameter which affects the atomization spray pattern, poor atomization and the particles size of fuel. The observed specific gravity of seed oils did not meet the Indian standards specifications for diesel fuel (0.820 to 0.860 at 15°C). So it was conformed by the earlier report by Peterson

et al. (1983) that the specific gravity of oils have to be reduced by splitting of glycerol from triglycerides of oil.

iv. Flash point

The flash point of diesel fuel was observed to be 51°C using the Pensky Martens apparatus. From the table 4.1 it was observed that the flash point of the selected tree seed oils was more than 200°C and maximum flash point was observed for neem oil (288°C). The flash point of coconut oil was almost closer to that of pungam oil. The lowest flash point was recorded for pinnay oil (211°C). The flash point of jatropha oil was 251°C which was lower than the values reported by Kandapal and Mira (1995) and Foidl *et al.* (1996). This may be due to the variation in fatty acid compositions in oil. The flash points of all selected tree seed oils were 4 to 5 times higher than the flash point of diesel fuel. The higher flash point indicates the involatile nature of oils. Goering *et al.* (1982) reported similar results for vegetable oils.

v. Cloud point

The cloud point of the selected tree seed oils ranged from 13 to 18°C whereas the cloud point of diesel fuel was 13°C. The cloud point of both pungam and pinnay oil was 15°C. The highest cloud point was observed for eluppai oil (18°C) and the lowest for jatropha oil (13°C). The cloud point of coconut oil was 4°C higher than the value of diesel fuel and the cloud point of palm oil was 16°C. The cloud point of all oils except jatropha oil was higher than diesel fuel.

vi. Pour point

The pour point of diesel fuel was observed as 1°C and the pour point of jatropha oil was 4°C which is 3°C higher than diesel fuel. The lowest pour point was recorded for neem oil (1°C) and the highest for coconut oil (13°C). The pour point of eluppai oil was 11°C, which is 10°C higher than diesel fuel. This is in good agreement with the findings of Goering *et al.* (1982) for vegetable oils. The higher pour point of tree seed oils than diesel fuel indicates poor flow characteristics of oil at lower temperatures.

vii. Carbon residue

The carbon residue of selected tree seed oils was observed between 0.45 and 0.64 per cent and for diesel fuel it was 0.21 per cent. The highest carbon residue for eluppai oil was 0.64 per cent, which was three times higher than diesel fuel. The carbon residue for jatropha oil was 0.54 per cent and neem oil recorded closer to jatropha oil. The lower carbon residue recorded for coconut oil (0.45%). From this it was observed that the selected tree seed oils did not meet the requirements of Indian standards for diesel fuel.

viii. Ash content

The ash content of selected tree seed oils was observed to vary between 0.011 and 0.021 per cent and the ash content of diesel fuel was 0.010 per cent. The maximum ash content was observed for eluppai oil and minimum for coconut

oil (0.011%). The ash content for jatropha oil was 0.013 per cent. The ash content of selected tree seed oils were slightly higher than that of diesel fuel.

4.2.2. Chemical properties

i. Free Fatty Acids (FFA)

The free fatty acid content of selected tree seed oils was determined by A.O.C.S method as described in the section 3.2.1. Higher FFA was observed for eluppai oil followed by pinnay, neem, and pungam oil. The lowest FFA was noted for coconut oil. FFA of palm and coconut oils were below 2 per cent. The variation of FFA from 1.44 to 17.30 per cent may be attributed to the rancidity of oil. The FFA of palm oil expressed in terms of palmitic acid (1.82 %) and for coconut oil it is expressed as luric acid (1.44 %). For other oils FFA is expressed in terms of oleic acid only.

ii. Acid value

The acid value of selected tree seed oils was calculated as 1.99 times of FFA and the variation was similar to the FFA results.

iii. Total glycerol content

The total glycerol content of oils varied between 12.36 to 15.03 per cent. The jatropha oil had higher total glycerol content of 15.08 per cent. The glycerol contents of pungam and coconut oil were 12.60 per cent. The glycerol content of pinnay, eluppai and palm oil ranged from 13.81 to 14.43 per cent. The total glycerol is the main cause of high viscosity and specific gravity of the oils.

iv. Free glycerol content

The free glycerol content for pinnay oil was observed as high (1.66 %) and no free glycerol content was observed in coconut and palm oils. The neem oil recorded a lower free glycerol content of 0.59 per cent.

v. Combined glycerol content

The combined glycerol content was calculated by finding difference between total and free glycerol content of the oils. Palm oil had high combined glycerol content of 13.45 per cent.

4.3. Production of biodiesel from selected tree seed oils

The major obstacle in using tree seed oils as alternate to diesel fuel in engines is higher viscosity and densities due to the presence of glycerol in triglycerides. The kinematic viscosities of tree seed oils as discussed earlier were found to be 6 to 11 times higher than diesel fuel viscosity. When diesel engines were operated with vegetable oils following were the major problems reported by Goering *et al.* (1982), Mori (1983), Pestes and Stanislaw (1984) Abdul (1985) and Bagby (1987).

- i. Fuel filter block
- ii. Severe engine deposits
- iii. Injector coking and piston ring sticking
- iv. Higher injection pressure and poor atomization

- v. Incomplete combustion due to poor fuel atomization and inefficient mixing with air

Heating the oils blending of oil with diesel fuel and transesterification methods were selected for this study as explained in section 3.3 in order to reduce the viscosity of selected tree seed oils.

A. Heating the oils

The viscosity of fuel is an important property for the proper operation of the injection system in diesel engines. To achieve viscosity of raw oils closer to viscosity of diesel fuel (4.86 cSt) an attempt was made to reduce the viscosity by heating the oils. The effect of temperature on kinematic viscosity of selected tree seed oils is shown in Figs. 4.1 to 4.7. It is evident that the kinematic viscosity of oils was decreased with increase in temperature. The trend of the curve was very close to straight line with R^2 value ranged from 0.798 to 0.988 (table 4.2). Abdul (1985) reported that oils may be preheated to achieve the desired viscosity. The viscosity value of oils indicated that the oil may be heated to 118 to 141°C to meet out the viscosity of diesel fuel at 40°C.

Bari *et al.* (2002) reported that palm oil temperature of 90°C did not damage the fuel injection system. It was concluded from the available studies that the higher heating oil temperature might have adverse effects on closely fitting parts

(plunger – barrel assembly) because the fuel injection was designed to supply fuel at ambient temperature to run the engines.

B. Blending of raw oil with diesel fuel

The experiment was conducted with four blended fuels viz., B₂₀, B₄₀, B₆₀ and B₈₀ and their kinematic viscosity of seed oils is shown in Figs. 4.8 to 4.14.

i. Pungam oil

The effect of blending on kinematic viscosity of pungam oil at 40°C is shown in Fig. 4.8. From the figure it was evident that increase in quantity of diesel fuel in the blended fuel decreased the viscosity of raw oil. The reduction in kinematic viscosity of blended fuels was from 42.70 to 5.36 cSt. The reduction in kinematic viscosity of blended fuels was 44 to 87 per cent due to addition of diesel fuel. The B₂₀ was recorded the lowest viscosity of 5.36 cSt which was 1.1 times higher than that of diesel fuel viscosity at 40°C.

ii. Palm oil

The effect of blending on kinematic viscosity of the blended palm oil fuels at 40°C is shown in Fig. 4.9. The kinematic viscosity of B₂₀ fuels was 85 per cent lower than raw oil viscosity. The per cent reduction in viscosity varied from 20 to 85 per cent which was the lowest reduction in kinematic viscosity of blended fuels among other blended oil fuels.

iii. Pinnay oil

The effect of blending on kinematic viscosity of the pinnay oil blended fuels at 40°C is shown in Fig. 4.10. The high reduction in viscosity of oil was achieved by blending of 80 per cent diesel fuel with 20 per cent raw oil (5.63 cSt). But this viscosity did not meet out the requirements of diesel fuel. The per cent reduction in kinematic viscosity varied from 34 to 87 for B₈₀ to B₂₀ oil fuels.

iv. Jatropha oil

Kinematic viscosity of different blending ratios of jatropha oil with diesel fuel is shown in Fig. 4.11. From the figure it was concluded that the kinematic viscosity decreased with increased blending amount of diesel fuel. The per cent reduction in kinematic viscosity of blended fuels was 44 to 86 for B₈₀ to B₂₀ ratios. Among four blending ratios B₂₀ recorded the lowest kinematic viscosity (5.25 cSt) and it was 1.1 times higher than recommended viscosity of diesel fuel.

v. Neem oil

The viscosity of B₂₀ fuel was observed as 5.51 cSt which was the lowest among other neem oil blends. But this viscosity was 1.13 times higher than that of diesel fuel viscosity at 40°C. The reduction in viscosity was varied from 28 to 85 per cent for B₈₀ to B₂₀ fuels (Fig. 4.12). Ziejewski *et al.* (1983) reported that engine even operated with blending fuel of sunflower oil and diesel fuel in the ratio of 1:3 (viscosity 4.88 cSt at 40°C), can not be recommended for long term use in

the direct injection diesel engines due to severe nozzle coking, sticking and thickening of lubricant oil.

vi. Coconut oil

The viscosity of B₈₀ and B₆₀ blends was sharply decreased with increasing amount of diesel fuel. The viscosity decreased gradually with increased amount of diesel fuel in the fuel (Fig. 4.13). The lowest kinematic viscosity of blended fuel was B₂₀ which was slightly higher (5.20 cSt) as compared with diesel fuel (4.86 cSt) at 40°C.

vii. Eluppai oil

The lowest kinematic viscosity of blend B₈₀ of eluppai oil was 26.84 cSt, which could not meet out the desirable viscosity of diesel fuel (Fig. 4.14). The lowest viscosity of eluppai oil blend was recorded for B₂₀ (5.63 cSt) and was high as compared with diesel fuel viscosity.

Bansal and Juneja (1989) studied short term test with neem oil blends in diesel engine. They reported that thermal cracking of neem oil blends took place earlier than diesel fuel which resulted in more smoke, particulates and spoiling of lubricants if the blend contained more than 30 per cent neem oil.

Early research work reported that the blended fuels even though it can be used in the place of diesel fuel created problem in diesel engines. Reducing the kinematic

viscosity of raw oil did not meet out the diesel fuel requirements. The viscosity of even B₂₀ blends of all seed oils was higher than viscosity of diesel fuel by 1 to 1.68 times. Peterson *et al.* (1983) reported that even 20 per cent blend was causing problems in engine like, thickening of lubricant oil, etc and not recommended for long-term engine operation.

C. Transesterification

The fuels obtained by the above methods *viz.* heating or blending were found to be unsuitable for diesel engines. The only solution to meet out the fuel standards for diesel engine is by converting the tree seed oils into methyl esters through transesterification process. The transesterification process produces the methyl ester from the tree seed oils by replacing methanol molecules in place of the glycerol molecules in triglycerides. It is a chemical reaction in which methanol and oils are mixed in the presence of catalyst to separate glycerol and methyl esters. The methyl ester produced from transesterification was named as biodiesel name itself describes the use of this fuel as an alternate to diesel fuel. The molecular weight of the oil required for transesterification reaction is the important parameter for biodiesel production. The molecular weight of oil was calculated based on composition of fatty acids in the triglycerides.

i. Fatty acid compositions

The gas chromatography with FID detector (GC-FID) was used to determine the fatty acid compositions of oils. The operating conditions used for GC-FID

detector was given in the section 3.3. The analyzed results of oils fatty acid compositions are depicted in the table 4.3. The saturated fatty acids like palmitic and stearic were found in all tree seed oils. The palmitic acid ($C_{16:0}$) was major saturated acid observed in the oils ranged from 12.9 to 30.6 per cent. The stearic acid ($C_{18:0}$) was the next predominant saturated acid in the oils and arachidic acid ($C_{20:0}$) was found to be less than five per cent in all tested oils. The oleic acid ($C_{18:1}$) was most dominating unsaturated component in the tested oils which had a range of 34.6 to 60.9 per cent except coconut oil. Linoleic acid ($C_{18:2}$) ranked as next unsaturated fatty acid in the oils next to oleic acid. The linoleic acid ($C_{18:2}$) observed in the jatropha oil was 31.6 per cent which recorded highest among other oils. Among the tree seed oils coconut oil showed a much variation in fatty acids composition, which had very high quantity of saturated fatty acids. The higher levels of saturated fatty acid can lead to freezing of oil at low temperatures, because of low melting points of saturated fatty acids.

Table 4.3. Fatty acids compositions of oils

Oil name	C_{16:0} %	C_{18:0} %	C_{20:0} %	C_{18:1} %	C_{18:2} %	Other acids, %	M W	TS FA	TU SA
Pungam	14.5	7.8	4.3	53.6	19.8	-	872	26.6	72.4
Pinnay	17.3	23.3	2.8	42.9	13.7	-	871	43.4	55.6
Palm	27.7	4.8	0.5	46.0	17.0	4	842	33.0	67.0
Jatropha	12.9	8.1	-	46.2	31.6	1.2	870	22.2	77.8
Neem	14.8	14.1	1.3	60.9	8.9	-	871	30.2	69.8
Coconut	30.6	3.5	0.05	34.6	0.5	60.35	668	-	-
Eluppai	25.5	26.6	3.2	32.5	12.2	-	866	55.3	44.7

ii. Biodiesel production from selected tree seed oils in a bench scale reactor

The free fatty acids of the selected tree seed oils ranged from 1.82 (palm oil) to 17.30 (eluppai oil) per cent (table 4.1). The acid value of selected tree seed oils ranged from 2.97 to 34.48.

Ma *et al.* (1999) noted that the glycerides should have an acid value less than one but in the present study the acid value of tree seed oils was found to be greater than one. However, Ma *et al.* (1998) suggested that more NaOH can be used to neutralize the free fatty acids (FFA) if the acid value was greater than one. Hence in the present study an excess amount of NaOH was used for transesterification reaction and the amount of NaOH used for different oils was presented in section 3.3.C.iii. The developed laboratory scale reactor was used in biodiesel production and water wash was done in separatory funnel (plates 4.1. and 4.2).

During the experiment of biodiesel production from pongam oil it was observed that the oil became wine red in colour when reacting with sodium methoxide solution. This may be due to presence of isolonchocarpin component in the oil which gave wine red colour after reacting with alkaline catalyst. The mixture thickened initially and became thin after 15 min. The colour of final product of reaction was light brown.

For the case of biodiesel production from the palm oil it was found that the oil became thick yellow colour after mixing with sodium methoxide solution. The mixture thickened within 15 min and became thin after 30 min. The colour of biodiesel from the palm oil was light yellow.

There was no transesterification reaction and only gel like formation occurred while experimenting with pinnay oil. The catalyst used in the reaction was 1.3 per cent. During the experiments oil and solution mixtures were mixed well and found to be thick after 3 min of reaction time. After 5 min the mixture became solid and gel like formation occurred. The higher FFA of pinnay oil led to soap formation during the transesterification reaction. Soap formed during transesterification of the FFA reacted with NaOH. In the reaction, the methanol reacted with oil and methyl esters were produced. At the same time water was also produced during the reaction, which reacted NaOH to yield the soap. Soap present in the reactant inhibited completion of the reaction, because sodium ions combined with free fatty acids present in oil. The soaps prevented the conversion of oil into biodiesel, which yielded gel like formation. The reaction was continued for 3 h and even then there was no change in gel composition. The gel formation was due to

higher free fatty acid content (9.30 %) of the oil. Similar results were obtained for biodiesel production from eluppai oil due to higher free fatty acid content (17.30%). The catalyst used in the reaction was 1.6 per cent for eluppai oil.

The above results are in conformity with that reported by Stumborg *et al.* (1996). They reported that the gel formation was due to high concentration of saturated fatty acid components, which yielded low conversion of triglycerides during the experiments. They also reported that the incomplete reaction caused the formation of mono-, and di-glycerides with high melting points forming a solid or gel like formation.

During biodiesel production the jatropha oil was converted into thicker mass after adding sodium methoxide solution. The thickened mixture became thin after 20 min. The oil and reactant mixture first became yellow in colour and after 15 min of stirring, the colour changed to light yellow.

In case of biodiesel production from the neem oil, it was noted that the oil turned into dark green in colour immediately after mixing with sodium methoxide solution. The mixture thickened and became thin after 30 min. The colour of the final product of reaction was light green.

The coconut oil became pale yellow colour after addition of sodium methoxide solution. The mixture became thin after 30 min. and yielded white coconut biodiesel.

Methyl ester conversion of oils ranged from 97.80 to 99.47 per cent. A maximum methyl ester conversion of 99.47 per cent was obtained from the jatropha oil. The methyl ester conversion for pungam and coconut biodiesels was nearly same (99.04 and 99.34%). The lowest methyl ester conversion was observed as 97.98 per cent for neem oils. The selected tree seed oils and their biodiesels are shown in Plate 4.3, 4.4, 4.5, 4.6 and 4.7.

iv. Methods adopted for biodiesel production from higher FFA oil

The methods used for biodiesel production from higher FFA oil (>7%) are explained in the section 3.3.C.iv. The results obtained from biodiesel production from pinnay and eluppai by using three methods is presented in the table 4.4. For the one and two step methods, the biodiesel was not produced and only gel like formation occurred. Pinnay and eluppai oils cannot be converted into biodiesel using NaOH catalyst due to the formation of soaps. The soaps were main cause to produce the mono, di-glycerides in the reaction, which yield the solid like formation of end products.

Table 4.4. Methods adopted for biodiesel production from higher FFA oil

S.No	Methods used	Methyl ester conversion, %	
		Pinnay	Eluppai
1	One step	-	-
2	Two step	-	-
3	Third method	84	71

In case of third method, the methyl ester conversions of the oils were found to be very low (71 to 84 %) as compared with low FFA oils (<7%). Biodiesel from the higher FFA oils like pinnay and eluppai is technically feasible but economically not feasible. Because of the very high (1:30 oil:methanol molar ratio) quantity of methanol required to yield better quality biodiesel. In general NaOH based transesterification was found to be not suitable for the biodiesel production from these oils. Acid based / pretreatment with acid to convert the FFA into biodiesel is the only solution to solve this problem.

4.4. Properties of biodiesels

The raw biodiesel was collected from separating funnel after removing glycerol and tested for the properties. The raw biodiesel was washed several times with water and allowed to settle as such for 3 hours to remove water and collected as pure biodiesel. The properties of raw and pure biodiesel produced from the seed oils are presented in table 4.5.

The pH value of all raw biodiesels was observed to be more than 7, which indicated the presence of alkaline catalyst in the raw biodiesel. The highest pH value was observed in palm biodiesel (9.6), pungam and neem biodiesel had same pH (8.7) and the lowest value of pH was for coconut biodiesel (8.2). The impurities such as glycerol, sodium hydroxide and unreacted methanol in biodiesel were removed by using water wash. The quantity of water to raw biodiesel used in washing was 3:1.

The sodium content in the raw biodiesel ranged from 42 ppm in jatropha biodiesel to 115 ppm in palm biodiesel, whereas it was found to be 1.2 and 2.3 ppm for pure palm and jatropha biodiesel, respectively. Higher sodium content was due to the presence of alkaline catalyst in the raw biodiesel. The sodium content in the raw biodiesels was reduced through water washing and the pH value was found to be 7 for all pure biodiesels.

The difference in specific gravity of raw and pure biodiesel was due to the presence of impurities like glycerol and NaOH and unreacted methanol in raw biodiesel. Among the raw and pure biodiesels palm biodiesel was noted with high specific gravity (0.8861 and 0.8850 respectively). The jatropha biodiesel recorded the lowest specific gravity (0.8630 and 0.8618 respectively).

Kinematic viscosity of raw biodiesels ranged from 5.01 to 5.94 cSt. After water washing, the kinematic viscosity of pure biodiesel ranged from 4.98 to 5.89 cSt due to less specific gravity and absence of impurities after washing.

A. Ultimate analysis

The elemental compositions of the tested biodiesels were presented in the table 4.6. The carbon content in the biodiesels ranged from 75.77 to 76.40 per cent by mass. The oxygen content in the biodiesel varied from 11.40 to 11.72 per cent (by mass) which can help in the complete combustion of the fuel in engine.

Table 4.6. The elemental composition of biodiesels

Sl. No.	Biodiesel used	Carbon, wt %	Hydrogen, wt %	Nitrogen, wt %	Oxygen, wt %	Sulphur, wt %
1	Pungam	76.40	12.09	<0.02	11.40	<0.04
2	Palm	76.12	11.94	<0.04	11.89	<0.02
3	Jatropha	76.33	12.11	<0.02	11.52	<0.02
4	Neem	75.77	12.45	<0.04	11.72	<0.02
5	Coconut	76.08	12.25	<0.04	11.61	<0.02

Also the higher oxygen content in the fuel will reduce its heating value. The hydrogen content ranged between 11.94 and 12.45 per cent. The compositions of biodiesel showed that the sulphur content was in trace amount (<0.02). From this data, it is clear that the emission of SO_x will be low compared to than conventional fuel.

B. Fuel Properties

The fuel properties of pure biodiesels are presented in the table 4.7.

i. Calorific value

The calorific values of biodiesel were slightly lower than the raw oils due to chemical changes after transesterification process in the fatty acid compositions of oils. The heat contents of biodiesel were approximately 10 to 14 per cent lower than that of diesel. The calorific values for biodiesel ranged from 38.11 to 39.81 MJ/kg. The maximum value was observed for pungam biodiesel (39.81 MJ/kg), low value for neem biodiesel (38.11 MJ/kg) and the calorific value of the jatropha biodiesel was 39.17 MJ/kg.

ii. Kinematic viscosity

In pongam oil the viscosity was reduced from 41.89 to 5.59 cSt after transesterification reaction and washing. The viscosity of biodiesel was found to be 1.15 times higher than viscosity of diesel fuel (4.86 cSt).

For palm biodiesel the viscosity was recorded as 5.89 cSt, which was 1.2 times higher than that of diesel fuel which to raw palm oil had 11 times more viscosity than diesel fuel (4.86 cSt).

The kinematic viscosity of jatropha biodiesel was found to be 5.32 cSt, whereas the viscosity of jatropha oil was 36.25 cSt.

After biodiesel production from neem oil the viscosity of neem biodiesel was reduced to 15 per cent of the viscosity of raw oil.

The kinematic viscosity of coconut biodiesel was reduced to 18 per cent of the viscosity of coconut raw oil through transesterification reaction. The viscosity of biodiesel was almost closer to viscosity of diesel fuel.

Among the produced biodiesels, the palm biodiesel recorded the highest viscosity (5.89 cSt) than other biodiesels. The coconut biodiesel was found to have minimum viscosity. As the viscosity of produced / tested biodiesels produced was very closer to that of diesel fuel (4.86 cSt) they can be used in diesel engine. The viscosity reduction in biodiesels noted as $1/5^{\text{th}}$ to $1/9^{\text{th}}$ of their raw oils. The viscosity of oil was equal to diesel viscosity only at temperature range of 118 to

141°C by heating the oil. But a highest reduction in viscosity of oils was achieved through transesterification process by separating the glycerol from the oils.

iii. Specific gravity

The specific gravity of biodiesels varied from 0.8618 to 0.8850 at 40°C, which was comparable to the DIN standards (0.875 to 0.890) whereas the specific gravity of original oils was observed as 0.92 61 to 0.9098. After transesterification, the specific gravity of biodiesel was lower than the oils due to removal of glycerol from the triglycerides. The specific gravity of biodiesel was found within the requirements of DIN standards. The palm biodiesel recorded a higher specific gravity than other biodiesels and specific gravity of pungam, neem and coconut biodiesels was found to be closer that of diesel fuel. The minimum specific gravity was noted for jatropha biodiesel (0.8618). The recommended specific gravity for diesel fuel was 0.820 to 0.860 at 15°C (IS 1460: 2000).

iv. Flash point

The flash point of biodiesels was noted as 3 to 3.5 times higher than diesel fuel flash point (51°C). The flash point ranged from 156 to 179°C and 211 to 288°C for biodiesels and their sources, respectively. The lower flash point temperature of biodiesel indicates an improvement in volatile property of the biodiesel. This may be due to the replacement of the glycerol by molecules of methanol in the fatty acid compositions of the biodiesel. The coconut biodiesel recorded a lower flash point temperature (156°C) and maximum for neem biodiesel (179°C). Even though the

flash points of these biodiesels as higher than diesel fuel, they meet out the DIN standards for biodiesels ($>110^{\circ}\text{C}$).

v. Cloud point

The cloud point of biodiesels was observed to be between 12 and 16°C and it was slightly higher than the raw oils. This may be due to changes in composition of biodiesels by transesterification reaction. The observed value of diesel cloud point was 13°C . The cloud point of jatropha biodiesel was found 1°C lower than diesel. The coconut biodiesel recorded a high cloud point value (16°C), which indicated formation of crystals in biodiesel at lower temperatures of testing. This biodiesel required additional heating during winter to improve flow behavior of the biodiesel.

vi. Pour point

A lowest pour point was observed for neem biodiesel (-1°C), which was 2°C lower than diesel fuel. The highest pour point was observed for coconut biodiesel (11°C). The coconut biodiesel was recorded the higher pour and cloud points due to its higher saturated fatty acid in oil composition. The jatropha biodiesel had pour point of 3°C which was 4°C for jatropha raw oil. The decrease in pour point of biodiesel may be due to variation in the fatty acid compositions.

vii. Carbon residue

The carbon residue is an important property for indicating the coking characteristics of fuel. The carbon residue for biodiesel must be lower than 0.50 per cent

(DIN Standards) to perform better in the engine. The observed carbon residue of biodiesels varied between 0.24 to 0.32 per cent. This range was found to be within the acceptable range of biodiesel standards. The lower carbon residue was recorded for jatropha biodiesel (0.24 %), which was 0.54 per cent for raw jatropha oil. The reduction in carbon residue indicates an improvement in coking characteristics of the fuel. The maximum carbon residue recorded for neem biodiesel was 0.32 per cent. The carbon residues of the pungam, palm and coconut biodiesels were 0.25, 0.30 and 0.28 per cent respectively. All the biodiesels produced in the present study met the DIN standards.

viii. Ash content

The ash content of biodiesel varied between 0.010 and 0.015 per cent.

The ash content of diesel fuel was 0.010 per cent. The ash content of jatropha oil was 0.013 per cent, which was reduced to 0.011 after transesterification.

The reduction in ash content was due to change made in compositions due to reaction and removal of glycerol from the oils. The lowest ash content was observed for coconut biodiesel and higher value for palm biodiesel.

C. Chemical properties

The chemical properties revealed about the impurities present in the biodiesel. The impurities like FFA, free glycerol and combined glycerol may spoil the fuel qualities. The chemical properties studied for biodiesel are discussed below.

i. Free fatty acids

The free fatty acids of the biodiesel were found to be 0.27 to 1.20 per cent for palm to neem biodiesel. The free fatty acid of their raw oils varied from 1.44 to 5.60 per cent. The free fatty acid of biodiesel was lower than raw oils. This may be due to neutralization of free fatty acids by excess amount of NaOH catalyst during biodiesel production process.

ii. Acid value

The acid value of produced biodiesels was lower than DIN biodiesel standards. The maximum acid value was obtained for neem biodiesel (2.87) and minimum for palm biodiesel (0.537). Biodiesel from edible oils such as palm and coconut had lower acid value than non-edible biodiesels.

iii. Total glycerol content

The observed total glycerol content of biodiesels varied from 0.08 to 0.25 per cent. The total glycerol content of jatropha and coconut biodiesels was found to be same as 0.08 per cent. The glycerol content of pungam, palm and neem biodiesel ranged from 0.12 to 0.25 per cent.

iv. Free glycerol

There was no free glycerol observed in palm, jatropha and coconut biodiesels. The neem biodiesel had a higher free glycerol content of 0.18 per cent.

The free glycerol content of the neem biodiesel was greater than the recommended free glycerol content (DIN biodiesel standards).

v. Combined glycerol

Palm biodiesel had a higher combined glycerol content of 0.17 per cent. The combined glycerol was same as total glycerol for palm, jatropha and coconut biodiesels, because of the absence of free glycerol observed in these biodiesels.

From the above results it was observed that all the properties of biodiesels studied were fulfilled the requirement of biodiesel DIN standards except for the total and free glycerol values of neem biodiesel and free glycerol values of pungam biodiesel.

4.5. Optimal conditions for biodiesel production

The FFA of the coconut oil was found to be lowest (1.60 %) among the other oils and neem recorded the highest value (5.33 %). The FFA of the oil was important parameter, which affects the biodiesel production by producing the soap during the transesterification reaction. The other properties were presented in the table 4.8. The specific gravity of the oils ranged from 0.9098 to 0.9265. Pinnay and eluppai oils were not considered for this study due to higher FFA content of the oils.

Table 4.8. The properties of raw oils

Sl. No.	Properties	Pungam oil	Palm oil	Jatropha oil	Neem oil	Coconut oil
1	Specific gravity	0.9210	0.9265	0.9124	0.9194	0.9213
2	Kinematic viscosity at 40°C, cSt	42.59	47.26	36.57	38.26	26.95
3	FFA, %	4.31	1.63	3.11	5.33	1.60
4	Acid value	8.58	3.24	6.19	10.61	3.20
5	Total glycerol, %	12.50	13.40	13.60	12.60	12.10

For this study three levels of molar ratios and catalyst amount were used to determine the optimal condition for better methyl ester conversion in biodiesel production. The experimental conditions for biodiesel production from selected oil were presented in section 3.5. The molar ratio of oil to methanol is important parameter in biodiesel production. For this study three levels of molar ratios (1:4.5, 1:6 and 1:7.5) were used.

A. Pungam biodiesel production

i. Effect of molar ratio

The effect of molar ratio of oil to methanol on pungam biodiesel production is shown in Fig. 4.15. In 0.1 M catalyst amount the methyl ester conversion sharply raised from 75.8 to 94.35 per cent by increase in molar ratio from 1:4.5 to 1:7.5. In case of 0.2 M catalyst amount conversions were observed as 79.70, 89.30 and 96.80 per cent at molar ratio of 1:4.5, 1:6 and 1:7.5 respectively. The methyl ester conversion at 1:6 found to be 7.5 per cent lower than molar ratio of 1:7.5. The excess methanol used in reaction led to better conversion with same catalyst

amount. The methyl ester conversions for pungam oil with 0.3 M catalyst amount were 82.30, 91.30 and 98.65 per cent at molar ratios of oil to methanol 1:4.5, 1:6 and 1:7.5 molar ratios respectively. The maximum methyl ester conversion was found to be 98.65 per cent for 1:7.5. From the study, it concluded that the molar ratio of 1:4.5 failed to yield better conversion (<85%). The molar ratio 1:7.5 performed better than 1:4.5 and 1:6.

ii. Effect of catalyst amount

At 1:4.5 molar ratio of oil:methanol, the methyl ester conversion increased from 75.80 to 82.30 per cent, as catalyst amount increased from 0.1 to 0.3 M. At 1:6 molar ratio, the methyl ester conversion raised from 86.60 to 91.30 per cent as catalyst amount increased from 0.1 to 0.3 M. For 1:7.5 molar ratio, the methyl ester conversion increased with molar ratio as well as increased amount of catalyst. The statistical analysis showed (table 4.9) a highly significant difference between molar ratio (M) and catalyst amount (C). The best conversion occurred at molar ratio of 1:7.5 with 0.3 M catalyst amount. The combinations like molar ratio of 1:4.5 with 0.1 M catalyst yielded a lowest conversion among the treatments.

Table 4.9. Analysis of variance for pungam biodiesel production

Source	Degrees of freedom	Sum of squares	Mean sum of squares	F value
Molar ratio (M)	2	1373. 56	686.78	23997.76**
Catalyst amount (C)	2	116.71	58.35	2039.13**
M × C	4	5.18	1.30	45.26**
Error	18	0.5 1 5	0.03	1.00
Total	26	1495.97	57.54	2010.49

B. Palm Biodiesel Production

i. Effect of molar ratio

The effect of molar ratio on palm biodiesel production is shown in Fig. 4.16. The methyl ester conversion depends upon the quantity of alcohol used in the transesterification reaction. The maximum conversions obtained for palm biodiesel production with 0.1 M were 94.65, 96.80 and 98.65 per cent for the molar ratio of 1:7.5. In case of 0.2 M catalyst amount, conversions for the reaction were recorded as 96.90, 99.15 and 99.70 per cent at molar ratio of 1:4.5, 1:6 and 1:7.5 respectively. The methyl ester conversion at 1:7.5 was found to be 3.20 and 0.55 per cent higher than that of molar ratio 1:4.5. It showed that the excess amount of methanol used in the transesterification reaction improved the conversion of oil to methyl ester. The maximum methyl ester conversion was found to be 99.55 per cent for 1:7.5. This value was closer to the molar ratio of 1:6 with 0.3 M catalyst amount. From the statistical analysis, it was concluded that as the molar ratio of oil to methanol was increased the methyl ester conversion increased. The molar ratio of 1:4.5 yielded less than 95 per cent of methyl ester conversion during the transesterification reaction. The molar ratio of 1:6 and 1:7.5 performed better than 1:4.5 and yielded the closer value of methyl ester conversions.

ii. Effect of catalyst amount

At 1:4.5 molar ratio of oil:methanol, the methyl ester conversion increased from 92.70 to 97.30 per cent, as catalyst amount increased from 0.1 to 0.3 M. At molar ratio of 1:6, methyl ester conversion increased from 96.90 to 99.55 per cent as the catalyst amount increased from 0.2 to 0.3 M. In case of molar ratio of 1:7.5, 0.3 M catalyst

speeded up the reaction and converted oil into biodiesel up to 99.55 per cent. In this experiment, the methyl ester conversion was increased 97.30 to 99.50 per cent for increased amount of catalyst from 0.1 to 0.3 M. From the statistical analysis, it was concluded that the methyl ester conversion at 1:7.5 with 0.1 and 0.2 M was on par with molar ratio of 1:6 with 0.3 M. So among the different combinations, the molar ratio of 1:6 with 0.3 M was found to be best for better yield of biodiesel from the palm oil.

Table 4.10. Analysis of variance for palm biodiesel production

Source	Degrees of freedom	Sum of squares	Mean sum of squares	F value
M	2	202.68	101.34	10431.53**
C	2	3.32	1.66	170.68**
M × C	4	3.85	0.96	99.09**
Error	18	0.17	0.01	1.000
Total	26	210.02	0.08	831.49

C. Jatropha biodiesel production

i. Effect of molar ratio

At 0.1 M catalyst amount, the methyl ester conversions were increased from 95.10 to 97.30 per cent with increase in molar ratio from 1:4.5 to 1:7.5 (Fig.4.17). It indicated that the methyl ester conversion was improved to 2.20 per cent by increasing methanol amount by 50 to 150 per cent in excess than stoichiometric quantity. The lowest methyl ester conversion observed for combinations of 1:4.5 molar ratio and 0.2 M catalyst amount was 2 per cent lower than that of 1:7.5. For the case of 0.3 M catalyst amount, the methyl ester conversion at of the molar

ratio of 1:6 and 1:7.5 was found to be 99.70. There was no effect on methyl ester conversion of jatropha biodiesel by increasing molar ratio from 1:6 to 1:7.5. From the study it was concluded that as the molar ratio of oil to methanol increased the methyl ester conversion increased. But there was no significant effect on methyl ester conversion of biodiesel by increasing molar ratio (1:6 to 1:7.5) at 0.3 M. The methyl ester conversion at 1:6 and 1:7.5 was found to be the same.

ii. Effect of catalyst amount

The methyl ester conversion at molar ratio of 1:4.5 was raised from 95.10 to 99.20 per cent due to increase in catalyst amount from 0.1 to 0.2 M. The methyl ester conversion for 1:6 molar ratio was recorded as 96.30, 99.50 and 99.70 for the catalyst amount 0.1, 0.2 and 0.3 M respectively.

Table 4.11. Analysis of variance for jatropha biodiesel production

Source	Degrees of freedom	Sum of squares	Mean sum of squares	F value
M	2	46.75	23.38	1086.69**
C	2	11.70	5.85	271.98**
M × C	4	2.85	0.71	33.10**
Error	18	0.39	0.021	1.00
Total	26	61.69	2.37	110.30

For 1:7.5 molar ratio, the methyl ester conversion increased with excess amount of methanol and catalyst. The per cent conversion for jatropha biodiesel was in closer range for molar ratio 1:7.5. The analysis of variance for jatropha biodiesel

production is depicted in the table 4.11. There was a highly significant difference between the molar ratios in the reaction. In the present study, the higher methyl ester conversion was observed for the molar ratio of 1:7.5, but the molar ratios of 1:6 with catalyst 0.3 M and 1:5 molar ratio with 0.2 M were found to be on par. The optimal conditions for jatropha biodiesel production were identified as molar ratio of 1:6 with 0.3 M catalyst amount.

D. Neem biodiesel production

i. Effect of molar ratio

The Fig.4.18 shows the influence of the methanol amount on conversion of neem biodiesel. The methyl ester conversion of the neem biodiesel increased with excess amount of methanol used in the reaction. The methyl ester conversions at 0.1 M catalyst amount was noted as 82.65, 86.65 and 89.45 per cent for molar ratios of 1:4.5, 1:6 and 1:7.5 respectively. Six per cent increase in conversion was achieved by increase in molar ratio from 1:4.5 to 1:7.5. In case of 0.2 M catalyst amount, conversion at molar ratio of 1:4.5 was 2 and 3 per cent lower than that of molar ratios of 1:6 and 1:7.5. The ester conversions at 0.3 M catalyst amount were found to be highest among the other combinations. The methyl ester conversion at this catalyst was high due to more amount catalyst used to neutralize the FFA present in the neem oil (5.33%).

ii. Effect of catalyst amount

From the figure it was clear that the methyl ester conversion increased with the increase in catalyst amount. The higher catalyst combinations performed better than the other catalyst levels because the FFA content of neem oil was 5.33 per cent.

The methyl ester conversion of the neem biodiesel at 0.1 M catalyst amount was low due to insufficient amount of the catalyst used to convert the FFA present in neem oil into biodiesel. The conversion at molar ratios of 1:6 and 1:7.5 with 0.3 M was found to be 9 to 11 per cent higher than that of 0.1 M catalyst amount. The analysis of variance for neem biodiesel production is presented in the table 4.12. There was highly significant difference between the factors. The methyl ester conversion at molar ratio of 1:6 and 1:7.5 with 0.3 M was found to be on par. The best conversion for neem oil into biodiesel was observed at molar ratio of 1:6 with 0.3 M catalyst amount.

Table 4.12. Analysis of variance for neem biodiesel production

Source	Degrees of freedom	Sum of square	Mean sum of squares	F value
M	2	591.29	295.64	19337.14**
C	2	77.68	38.84	2540.48**
M × C	4	19.58	4.89	320.13**
Error	18	0.28	0.02	1.00
Total	26	688.82	26.49	1732.84

E. Coconut biodiesel production

i. Effect of molar ratio

The methyl ester conversion for coconut biodiesel with three different molar ratios showed that the amount of methanol used had a significant effect on biodiesel quality. The methyl ester conversion increased as the quantity of alcohol increased (Fig. 4.19). During the transesterification reaction for coconut oil with 0.1 M catalyst amount, the methyl ester conversion was increased from 92.30 to

99.04 for the molar ratio from 1:4.5 to 1:7.5. In case of 0.2 M catalyst amount, the conversion was observed as 1.4, 6.2 and 0.6 per cent higher than that of molar ratio of 1:4.5, 1:6 and 1:7.5 respectively, obtained with 0.1 M. The methyl ester conversion at 1:7.5 found to be 1 and 1.3 per cent higher than molar ratio of 1:4.5. For the case of 0.3 M catalyst amount, the methyl ester conversions were 94.80, 99.56 and 99.71 per cent for 1:4.5, 1:6 and 1:7.5 molar ratios respectively.

ii. Effect of catalyst amount

The completeness of the reaction was observed at molar ratios of 1:4.5, 1:6 and 1:7.5 with catalyst amount 0.1 to 0.3 M. The methyl ester conversion increased from 92.30 to 99.04, 93.70 to 99.37 and 94.80 to 99.71 per cent respectively. From this study it was concluded that the biodiesel conversion was increased with increase in the catalyst amount as well as in molar ratio. The best biodiesel production conditions for coconut were molar ratio of 1:6. There was highly significant difference between molar ratio and catalyst amount. The molar ratio of 1:4.5 with 0.1 M had poor performance.

Table 4.13. Analysis of variance for coconut biodiesel production

Source	Degrees of freedom	Sum of squares	Mean sum of squares	F value
M	2	199.73	99.87	62129.38
C	2	7.06	3.53	2195.47
M × C	4	4.27	1.07	664.70
Error	18	0.29	0.02	1.000
Total	26	211.09	8.12	5051.02

4.5.1. Ranking of biodiesels based on properties

The ranking of biodiesels is given in the table 4.14. It was rooted that jatropha and pungam biodiesels possess all the desirable properties next to coconut biodiesel. For countries like Philippines, which are major coconut oil producing country offered to use coconut oil (Machacon *et al.*, 2001) but in India it is an edible oil much in demand and cost of oil is very high. Hence, jatropha and pungam were selected for further study.

Table 4.14. Ranking of biodiesels based on fuel properties and standards (DIN)

Parameters	Rank			Remarks
	I	II	III	
Calorific value, MJ/kg	Pungam	Palm and Jatropha	Others	11 per cent less than diesel fuel
Kinematic viscosity, cSt	Jatropha and Coconut	Pungam	Others	1.5 times higher than diesel fuel
Specific gravity	Jatropha	Pungam and Neem	Others	
Flash point, °C (>110°C)	Coconut	Pungam and Palm	Jatropha and Neem	23°C more than coconut
Cloud point, °C	Jatropha	Pungam and others	Coconut	-
Pour point, °C	Neem	All others	Coconut	3°C more than neem for cold starting
Carbon residue%	Jatropha, and Pungam,	Coconut	Palm and Neem	All meet standards
Ash content, % (0.01 % for diesel fuel)	Coconut and Jatropha	Pungam	Neem and Palm	All meet standards
FFA, %	Palm, Coconut and Jatropha	Pungam	Neem	All meet standards

Acid value, (<5)	Palm, Coconut and Jatropha	Pungam	Neem	All meet standards
Total glycerol, % (<0.25 %)	Jatropha and Coconut	Pungam	Palm and Neem	All meet standards

4.5.2. Optimal biodiesel production for pungam and jatropha oils

As stated above, pungam and jatropha oils were selected to find out the optimal conditions for higher yield of biodiesel production. The laboratory scale reactor was used for this study. For this study each experiment replicated two times and average value of the methyl ester conversion has been reported. The number of experiment conducted were $2 \times 3 \times 3 \times 4 \times 2$ (144) for each oils. The parameters considered for the study of biodiesel production are given below.

a. Catalyst amount

Three levels of catalyst amount (by molar weight basis)

C_1 - 0.1; C_2 - 0.2; C_3 - 0.3

b. Molar ratio

Methanol: oil ratios were used to study their effect on biodiesel yield.

M_1 - 4.5:1; M_2 - 5.25:1; M_3 - 6:1

c. Reaction Time

Four reaction time (h) levels were used.

RT₁ - 0.5; RT₂ - 1.0;

RT₃ - 1.5; RT₄ - 2.0

A. Pungam biodiesel production

i. Effect of molar ratio

The molar ratios of oil to methanol used for the study were 1:4.5, 1:5.25 and 1:6. The methyl ester conversion at 1:4.5 molar ratio gave an unsatisfactory results (<80%) as compared with other molar ratios. The methyl ester conversion at molar ratio of 1:6 was noted as 89.34 to 97.81 and 92.50 to 98.55 per cent for catalyst amount of 0.2 and 0.3 M respectively. The methyl ester conversion increased as the molar ratio increased. The methyl ester conversion of 95.44 per cent was obtained at a molar ratio of 1:5.25 for 2 h, whereas the same conversion was obtained at 1.5 h with 0.2 M catalyst at a molar ratio of 1:6 (Figs. 4. 20 and 4. 21).

ii. Effect of catalyst amount

The catalyst amounts of 0.1, 0.2 and 0.3 M were selected for this study. The catalyst amount of 0.1 M yielded less than 85 per cent conversion. The methyl ester conversion increased with increasing amount of catalyst. At molar ratio of 1:5.25, the methyl ester conversion increased with an increase in the amount of catalyst from 0.2 to 0.3 M.

iii. Effect of reaction time

Four levels of reaction time such as 0.5, 1.0, 1.5 and 2 h were used for the study. The methyl ester conversion increased with increasing in reaction time. The conversion at the molar ratio of 1:5.25 with catalyst amount of 0.2 M increased from 86.40 to 95.44 per cent as reaction time increased from 0.5 to 2 h.

The methyl ester conversion at molar rate of 1:6 was increased from 89.34 to 97.81 and 92.50 to 98.35 per cent for increasing reaction time of 0.5 to 2 h for 0.2 and 0.3 M catalyst. The maximum conversion was obtained as 98.35 for 2 h reaction time with 1:6 molar ratio and catalyst amount of 0.3 M.

Analysis of variance for pungam biodiesel production is depicted in the table 4.15. From the statistical analysis, it was concluded that, highly significant difference existed between all the factors.

Table 4.15. Analysis of variance for pungam biodiesel production

Source	Degrees of freedom	Sum of square	Mean sum of squares	F - value
Molar ratio (M)	1	74.24	74.23	1068.35**
Catalyst amount (C)	1	26.17	26.17	376.65**
Reaction time (T)	3	318.34	106.11	1527.10**
M × C	1	4.00	4.00	57.63**
C × T	3	0.76	0.25	3.64*
M × T	3	9.19	3.06	44.08**
M × C × T	3	1074	3.58	51.53**
Error	16	1.11	0.07	1.00
Total	31	444.56	14.34	206.37

The molar ratio of 1:6, the catalyst amount of 0.3 M and the reaction time 2 h was found to be best (98.35 % conversion). These combinations were significantly different from all other combinations at one per cent level. For the combination of catalyst amount and reaction time, 0.3 M $\times$ 2 h (98.35 % conversion) was the best. The combination like 1:6 molar ratio $\times$ 0.2 M catalyst amount $\times$ 2 h reaction time (97.81 % conversion) was found to be best. In total a molar ratio of 1:6 $\times$ 0.3 M catalyst amount $\times$ reaction time of 2 h (98.35) was found to be the best. Based on experimental results, the optimal conditions for pungam biodiesel production was found to be a molar ratio of 1:6, catalyst amount of 0.3 M and reaction time of 2 h.

B. Jatropha biodiesel production

i. Effect of molar ratio

The molar ratio of oil to methanol is an important parameter in biodiesel production. The methyl ester conversion to biodiesel from jatropha oil for three molar ratios is given in Figs. 4.22 to 4.24. For all cases, the methyl ester conversion increased with increase in molar ratios. Best conversion occurred at a molar ratio of 1:6 due to 100 per cent excess of methanol used in the reaction. The theoretical minimum molar ratio required for transesterification was 1:3 of oil to methanol.

The methyl ester conversion for the molar ratios of 1:4.5, 1:5.25 and 1:6 was 65.56 to 80.22, 82.70 to 90.00 and 84.00 to 99.20 per cent respectively for minimum to maximum reaction times at varying catalyst amount.

ii) Effect of catalyst amount

Three catalyst amounts namely, 0.1, 0.2 and 0.3 M of sodium hydroxide were selected for this study. For each reaction, reaction temperature was maintained at 60°C and reaction time 0.5 to 2 h used with three different molar ratios.

The effect of catalyst amount on methyl ester conversion for three molar ratios is shown in Figs. 4.22 to 4.24. From the figures, it was observed that the methyl ester conversion increased with the increase in catalyst amount. At 1:4.5 molar ratio of oil:methanol (2 h), the methyl ester conversion increased from 78.75 to 81.32 per cent, as catalyst amount increased from 0.1 to 0.2 M. At molar ratio of 1:4.5 with a catalyst amount of 0.3 M, the methyl ester conversion was 75.82 to 80.22 per cent whereas it was found to be 78.02 to 81.32 per cent at 0.2 M of catalyst amount. At 1:5.25 molar ratio of oil: methanol, the methyl ester conversion ranged from 87.70 to 90.00 per cent as catalyst amount increased from 0.1 to 0.3 M.

For 1:6 molar ratio of oil to methanol, the methyl ester conversion increased with molar ratio and with increased amount of catalyst. In this experiment the methyl ester conversion at 2 h was increased 96.74 to 99.20 per cent for increased amount of catalyst from 0.1 to 0.3 M.

iii. Effect of reaction time

The reaction time of 0.5, 1, 1.5 and 2 h were used for all combinations of molar ratio and catalyst amount selected for the study. The conversion observed for molar ratio of 1:4.5 varied from 78.02 to 81.32 per cent as reaction time

increased from 0.5 to 2 h. The methyl ester conversion (1:5.25 molar ratio) of 87.70 per cent was obtained for 0.2 M catalyst, which was 88.30 per cent with 0.3 M at 1.5 h due to increase in catalyst amount. At molar ratio of 1:6, the methyl ester conversion increased with time at all catalyst levels. The maximum conversion was obtained at molar ratio of 1:6 and catalyst amount of 0.2 and 0.3 M.

Table 4.16. Analysis of variance for jatropha biodiesel production

Source	Degrees of freedom	Sum of square	Mean sum of squares	F - value
M	2	2930.16	1465.08	6794.31**
C	2	225.18	112.59	522.13**
T	3	659.01	219.67	1018.73**
M × C	4	53.55	13.39	62.09**
C × T	6	36.14	6.02	27.94**
M × T	6	116.13	19.36	89.76**
M × C × T	12	70.90	5.91	27.40**
Error	36	7.76	0.22	1.00
Total	71	4098.85	5773.00	267.72

Among the three molar ratios, the best conversion found at 1:6 molar ratio with 0.2 M of catalyst amount at a reaction time of 2 h. The methyl ester conversion increased with increasing amount of catalyst and reaction time for all the cases of molar ratios.

The analysis of variance for jatropha biodiesel production showed in the table 4.16. From the statistical analysis it was concluded that highly significant difference was found in the all the factors studied like molar ratio, catalyst amount and reaction time. Among the three different molar ratios, the molar ratio of 1.6

(92.81) was found to be the best. With reference to catalyst amount 0.3 M (87.16) was best. Among the four different reaction times 2 h (87.42) was noted as the best.

Among the different interaction combination between molar ratio and catalyst 1:6 and 0.3 M (94.58) was found to be the best. In catalyst amount and reaction time interaction both 0.2 M $\times$ 2 h (90.00) and 0.3 M $\times$ 2 h (87.16) were found to be the best. Both of them were on par with each other. With respect to molar ratio and reaction time interaction the molar ratio of 1:6 and 2 h (98.34) reaction time combination was observed as the best.

In the three factor interaction studies, the combination of both 1:6 molar ratio $\times$ 0.2 M catalyst amount $\times$ 2 h reaction time (99.15) and 1:6 molar ratio $\times$ 0.3 M catalyst amount $\times$ 2 h reaction time (99.20) were found to be the best. Both the factor combinations were on par with each other. From the study the optimal conditions for best yield of biodiesel from jatropha oil was found to be a molar ratio of 1:6, catalyst amount of 0.2 M and reaction of 2 h. These optimal conditions were selected for biodiesel production from jatropha oil in pilot biodiesel plant.

C. Analysis of oil and biodiesel in HPLC

HPLC makes feasible direct solution of all biodiesel components without derivatization. However acglycerols and methyl esters do not absorb in the UV region at wavelengths higher than 220nm, which causes detection problems (Michal Holcepek *et al.*, 1999). There are few reports of HPLC applied to biodiesel analysis. This allows detection of mono and diesel-glycerols as well as methyl esters as classes of components. This system is useful for quantifying the

various conversions of transesterification reactions and one for finger printing oil and esters.

Shimadzu LC-6A HPLC was used to analysis raw oil and their corresponding biodiesel and commercial diesel in order to compare various components qualitatively. In general, raw oil consists of a mixture of low and high molecular weight components spread over different reaction time in the chromatograph with higher molecular mass being detected at higher retention time. Commercial diesel has all its components clustered at lower retention time in its chromatograph. The oil, biodiesel and diesel were analyzed in HPLC and the chromatographs are given in the Annexure. In the chromatograph the esters appear in the order of increasing number of carbon atoms and of increasing unsturation levels for the same number of carbon atoms.

In case of diesel two predominant peaks were obtained at lower retention time whereas in oil and biodiesel more number of peaks corresponding to saturated and unsaturated fatty acids ere detected. Comparing raw oil biodiesel the chromatograph clearly showed that there is a shift of components present at higher retention time being absent in biodiesel and the presence of low molecular components in the biodiesel. This clearly indicates that by transesterification process higher molecular components are broken down to low molecular weight components which are identified low retention time in the chromatograph and tend to towards the chromatograph of diesel. From the chromatographs it was observed

that retention time of biodiesel components were less than raw oils due to reduction in molecular mass of individual components of the biodiesel due to transesterification process.

4.6. Biodiesel pilot plant

There is wide scope for biodiesel production from untapped oil sources such as pongamia and jatropha oils. Even though the optimal conditions were established for higher biodiesel yield for pongamia oil the free glycerol of pongamia biodiesel did not meet the DIN biodiesel standards. The pongamia oil required more reaction time to remove the free glycerol. Jatropha biodiesel having similar fuel properties of pongamia biodiesel except calorific value and flash point. Pongamia oil was not selected for large scale biodiesel production because of cost of oil was higher than the conventional fuel and lack of oil availability. Apart from this the pongamia oil had disagreeable odour and its yellowish orange colour change and darkens in storage whereas the jatropha oil having pleasant odour (castor smell) and colourless into yellow on storage.

Even after transesterification reaction the disagreeable odour from pongamia biodiesel was not completely removed. The removal of odour from the pongamia biodiesel needs additional treatment and it increases the cost of biodiesel. So the jatropha oil was selected and used in pilot plant for biodiesel production. The added advantages of jatropha plants were rapid growth, easy propagation and life span of 50 years (Pramanik, 2002).

In a process developed by Foidl *et al.* (1996) biodiesel production from jatropha oil the solution of methanolic KOH was added in two steps with a total reaction time of 11 h and in this process the reaction time is found to be high.

The methodology developed for present study is very simple for production of biodiesel from jatropha oil. The jatropha oil was selected for biodiesel production in the biodiesel pilot plant (Plate 4.8) and the produced jatropha biodiesel was used in diesel engine for its performance. Three experiments were conducted for biodiesel production from jatropha oil by pilot biodiesel plant.

4.6.1. Components

The catalyst used in the transesterification reaction was sodium hydroxide, which react with metals. So care was taken in the selection of raw materials for biodiesel plant fabrication. The biodiesel pilot plant was fabricated with stainless steel and plastics materials to avoid the corrosion problem. The components of the pilot plant are given in table 4. 17.

Table 4.17. Components of pilot biodiesel plant

S.No.	Name of the components	Units	Capacity (lit /unit)
1	Biodiesel reactor	1	70
2	Chemical mixing tank	1	25
3	Oil lifting pumps	2	-
4	Glycerol settling tank	3	50
5	Glycerol tank	1	50
6	Biodiesel washing tank	1	100
7	Air compressor	1	
8	Pure biodiesel storage unit	1	250

9	Control panel for monitoring the plant operation	1	-
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4.6.2. Biodiesel reactor

The biodiesel reactor consists of mechanical stirrer, oil lifting pumps, chemical mixing tank and steam generator. The biodiesel reactor was made of stainless steel material and completely insulated to arrest heat loss and maintain the reaction temperature during the plant operation. The function of mechanical stirrer was to make vigorous stirring of reactant mixtures. A steam generator was used to supply heat to the reactor to maintain reaction temperature. Oil lifting pumps were used to pump the raw oil to the reactor and also pump the reactant mixtures to glycerol settling tank. The chemical mixing tank having a stirrer is used to dissolve the catalyst in methanol. The chemical mixing tank was connected to biodiesel reactor with PVC pipe and gate valve arrangements.

4.6.3. Glycerol settling tank

The glycerol-settling tank was used to separate the glycerol from biodiesel. After transesterification reaction, the biodiesel and glycerol mixture were sent to the settling tank. The glycerol was settled by density difference between biodiesel and glycerol. The glycerol settled at bottom of the tank and biodiesel suspended at top of the glycerol layer. A gate valve was provided at the bottom of settling tank to remove glycerol from the glycerol settling tank. A pipe outlet for decanting biodiesel from the tank, was arranged at 10 cm above the bottom of the settling tank.

4.6.4. Biodiesel washing tank

The biodiesel washing tank was made of GI sheet with a capacity of 200 lit. It was placed below the glycerol settling tank. The crude biodiesel from glycerol settling tank was allowed to flow into the biodiesel washing tank. Air pipe was fixed at the bottom of washing tank and was connected to air compressor by pipeline. The air bubbles formed were used to mix crude biodiesel with water and wash crude biodiesel to remove catalyst and glycerol. A biodiesel drain out let was fixed at 50 cm above the bottom of the washing tank. The water was filled up to biodiesel outlet level before the supply of crude biodiesel from glycerol settling tank. The wastewater out let was fixed at bottom of the settling tank to drain out wastewater from each washing. The pure biodiesel was collected and stored in storage tanks. A control panel was used to monitor the plant operations during the jatropha biodiesel production.

4.6.5. Biodiesel pilot plant operation

Before starting up, the whole system was checked for presence of water which may affect transesterification reaction and biodiesel yield. The raw oil was pumped into the biodiesel reactor by oil lifting pump. The steam generator was put on and the steam was supplied to the reactor. The steam flow rate was regulated to maintain the reaction temperature. The NaOH pellets catalyst and methanol were mixed thoroughly in the chemical mixing tank. The solution of sodium methoxide was formed and was ready to be supplied to the reactor.

When the oil reaching the reaction temperature sodium methoxide solution was supplied to the biodiesel reactor. The stirrer was operated for optimum reaction time for proper mixing of reactant. Reaction temperature was monitored regularly and care was taken to maintain the reaction temperature at 60°C. After the completion of reaction time the product mixture was collected in the collecting tank. The lift pump was used to pump the product mixture to the glycerol settling tank. The product mixture in the glycerol settling tank was allowed to settle for 5 h for glycerol separation. The crude biodiesel was collected in the washing tank.

Air was supplied into the washing tank and air bubbles were formed. The small air bubbles were released from air vent which cause agitation. The agitation by air bubbles was led to wash crude biodiesel with water. The air bubbles reaching top of methyl ester layer were burst and released small amount of water carried by the air bubbles. These water molecules tend to move downwards and thereby effecting more washing. The washing time for each process was 2 h and washed biodiesel and wastewater were allowed for settling for an hour. Due to lesser density washed biodiesel was suspended at top layer over wastewater. The pure biodiesel was decanted from the washing tank and stored in container. The final product biodiesel was easily identified by distinct change of color from dark yellow to yellowish color. The jatropha biodiesel and its by product sample is shown in the plate 4.9. The total processing time required for biodiesel production from 50 lit of jatropha oil was 8 h. The time required was 10 h for 100 lit biodiesel

production in first day and second day onwards plant production was increased to 250 lit biodiesel per day (5 batches per day).

4.6.6. Material balance in pilot biodiesel plant

The amount of raw material, catalyst and methanol were measured before the biodiesel production process and end products such as glycerol and biodiesel are measured after the reaction. Final product of biodiesel was also measured after water washing. The mass balance was worked out as detailed below:

The input to reactor, $y = x_1 + x_4$

where, $x_1 = (x_2 + x_3)$

and

y – Total amount of reactants in the reactor, kg (input)

x_1 – Quantity of sodium methoxide solution, kg

x_2 – Quantity of NaOH, kg

x_3 – Quantity of methanol, kg

$$y = x_1 + x_4$$

$$x_1 = x_2 + x_3$$

$$= 0.12 + 2.88$$

$$= 3.00 \text{ kg}$$

The input to glycerol settling, $y_1 = y$

Out put from the glycerol settling tank, $y_2 = y_3 + y_4$

where,

y_3 – Quantity of glycerol (2.25), kg

y_4 – Quantity of biodiesel crude (15.75), kg

$$y_3 = 2.25 \text{ kg}$$

Input to the washing tank, (y_5)

$$\begin{aligned} y_5 &= y_4 + x_5 \\ &= 15.75 + 15 \\ &= 30.75 \text{ kg} \end{aligned}$$

where

x_5 – quantity of water used, kg

Out put from washing tank

$$y_6 = y_7 + y_8 + y_9$$

y_7 – quantity of pure biodiesel, kg

y_8 – quantity of waste water generated, kg

y_9 – quantity of mixture of methyl ester, glycerol and untreated oil layer, kg

$$\begin{aligned} y_6 &= y_7 + y_8 + y_9 \\ &= 14.90 + 15 + 0.85 \\ &= 30.75 \text{ kg} \end{aligned}$$

Ingredients / Batch

$$\text{Oil} = 15 \text{ kg}$$

$$\text{Methanol} = 2.88 \text{ kg}$$

$$\text{NaOH} = 0.12 \text{ kg}$$

Out put per batch

$$\text{Pure biodiesel} = 14.90 \text{ kg}$$

$$\text{Glycerol} = 2.25 \text{ kg}$$

Layer of mixtures = 0.85 kg

4.6.7. Fuel properties of jatropha biodiesel blended fuels

The biodiesel produced from jatropha oil in the biodiesel pilot plant was used for engine testing. The four levels of blending of jatropha biodiesel with diesel fuel viz., B₂₀, B₄₀, B₆₀ and B₈₀ and jatropha biodiesel were chosen for this study and compared with diesel fuel. The fuel properties of selected fuels from jatropha biodiesel and diesel fuel are presented in the table 4.18.

Table 4.18. Fuel properties of jatropha biodiesel blended fuels

Properties	B ₀	B ₂₀	B ₄₀	B ₆₀	B ₈₀	B ₁₀₀
Specific gravity	0.8396	0.8437	0.8482	0.8530	0.8576	0.8621
Kinematic viscosity at 40°C, cSt	4.86	4.96	5.03	5.14	5.26	5.37
Calorific value, MJ/kg	44.42	43.25	42.072	41.139	40.174	39.174
Flash point, °C	51	55	76	98	109	174
Carbon residue, %	0.21	0.21	0.22	0.22	0.24	0.24

From the table 4.18 it was concluded that the influence of diesel amount in the blended fuels was very high in reducing the specific gravity of the jatropha oil blended fuels. In jatropha biodiesel blended fuels increase in amount of diesel fuel showed no effect on specific gravity of fuels. Even though the jatropha raw oil blended fuels met the requirement of diesel fuel standards, the viscosity of these fuels was higher than the desirable diesel fuel viscosity. The kinematic viscosity of

jatropha biodiesel blended fuels increased with increase in amount of biodiesel level. The viscosity of B₂₀ biodiesel blended fuel was 4.96 cSt which was almost closer to desirable viscosity of diesel fuel (4.86 cSt). The blending of jatropha raw oil with diesel fuel reduced the viscosity from 36.25 to 5.25 cSt. But the jatropha biodiesel alone brought down the viscosity from 36.25 to 5.37 cSt. When it was blended in the diesel fuel the kinematic viscosity of B₂₀ fuel was the lowest (4.96 cSt). The viscosities of biodiesel blended fuel have little effect on reducing the viscosity of biodiesel. The calorific value of blended fuels was improved by addition of diesel fuel in the biodiesel blends. The heating value was increased with decrease in biodiesel amount and it ranged from obtained for B₈₀ to B₂₀ fuel. The blended fuels had effect in reducing the flash point of the jatropha oil blended fuels, which indicated the improvement in the volatile nature of the fuels. The flash point of blended fuels was increased with increase in amount of biodiesel in the fuels. Similar trend was observed for carbon residue of blended fuels. The fuel properties of jatropha biodiesel blended fuels met the diesel fuel and biodiesel standards.

4.7. Performance of biodiesel fuelled engine

A Kirloskar diesel engine of 5.2 kW capacity was used in the experiments without any modifications. The diesel engine and electrical loading unit are shown in Plates 4.10 and 4.11. The diesel engine coupled with AC alternator was used to conduct the engine performance testing with *Jatropha curcas* biodiesel and its

blends with diesel fuel. The details of fuels used for engine testing was given in the section 3.6.

i. Fuel consumption

The fuel consumption of engine was increased with increase in amount of biodiesel blends (Fig. 4.25). In the case of jatropha biodiesel the fuel consumption was about 14 per cent higher than that of diesel. This may due to higher specific gravity and lower calorific value of the biodiesel fuel as compared with diesel fuel. The calorific value of the jatropha biodiesel was about 12 per cent lower than that of diesel fuel. The per cent increase in fuel consumption of biodiesel blends (B₂₀ to B₈₀) ranged from 2 to 13 per cent than diesel fuel due to increase in calorific value of these fuels. This in conformity with similar results reported by Monyem (1998) and Silvio *et al.* (2002).

ii. Speed

The speed of the engine when operating with tested fuels is depicted in the table 4.19. Using a hand held tachometer the speed of the camshaft was measured and the speed of crankshaft was calculated by doubling the value of camshaft speed. At tested load conditions the rpm of engine ranged from 1490 to 1526 for all fuels. It showed that there was not much variation in speed on different fuels.

iii. Power out of the engine

The power output of the engine was calculated from the electrical output of alternator. The power output of the engine on jatropha biodiesel blends at different

loads is depicted in the table 4.20. From the results it was observed that there was no significant difference between biodiesel and its blends as compared with diesel.

At 2 kW load power output of the engine in diesel mode was 2.03 kW (3.41 hp), whereas it was 2.05 kW (3.44 hp) for biodiesel alone. From this study it was observed that power output of the engine did not change with jatropha biodiesel. The jatropha biodiesel performed similar to that of diesel fuel in diesel engine. The power output of the engine varied from 2.02 to 2.05 kW (3.39 to 3.47 hp) for jatropha diesel blended fuels. Among the blended fuels the B₆₀ fuel was found to be slightly higher than other fuels.

The power output of the engine operated with diesel fuel at 2.5 kW load condition was 2.53kW (4.24 hp) whereas it was 2.56 kW (4.29 hp) for biodiesel alone. The power output of the engine with jatropha biodiesel was similar to diesel fuel; but the consumption of the jatropha biodiesel was higher because of the calorific value of the jatropha biodiesel was 11 per cent lower than diesel. At this load condition power output of the engine for blended fuels ranged from 2.51 to 2.57 kW (4.20 to 4.31 hp).

In case of 3.5 kW load condition, the power output of the engine with biodiesel fuel mode was recorded as 3.58 kW (5.99 hp), whereas it was 3.55 kW (5.95 hp) for diesel fuel mode. The power output of the engine varied from 3.55 to 3.57 kW (5.95 to 5.98) for jatropha biodiesel blends.

In general it can be concluded that there was no difference between jatropha biodiesel and its blends and diesel.

iv. Specific fuel consumption

The specific fuel consumption was calculated by fuel consumption divided by the rated power output of the engine. The results of specific fuel consumption followed the similar trends of fuel consumption (Fig 4.26). The per cent increase in specific fuel consumption ranged from 3 to 14 for B₂₀ to B₁₀₀ fuels. The range of increase in consumption was found to be similar under all load conditions. The per cent increase in specific fuel consumption was increased with decreased amount of diesel fuel in the blended fuels. This may due to lower heating value of the fuels and higher mass of fuel flow to meet out the engine loads.

v. Brake thermal efficiency

According to Canakci and Van Gerpan, (2001) brake thermal efficiency is defined as actual brake work per cycle divided by the amount of fuel chemical energy as indicated by lower heating value of fuel. The brake thermal efficiency for tested biodiesel and its blends was found to be slightly higher than that of diesel fuel at tested load conditions (table 4.19). For diesel fuel it varied between 28.64 to 33.01 per cent. There was no difference between the biodiesel and its blended fuels efficiencies. The brake thermal efficiencies of engine when operating with biodiesel were 28.84, 30.58 and 33.12 per cent at 2, 2.5 and 3.5 kW load conditions. The brake thermal efficiency of biodiesel was found to be 0.11 to 0.28 per cent higher than diesel fuel. From this it concluded that the engine was efficient with both jatropha biodiesel fuels

and diesel fuel. This is in accordance with results reported by Yahya (1988), Chang and Van Gerpan (1997) and Monyem (1998).

vi. Emission profile

The exhaust emission from the engine was monitored with KM900 exhaust gas analyzer and results are discussed briefly as given below.

a. Excess air utilization

The excess air utilization for different fuels was monitored by KM900 exhaust gas analyzer. For all tested fuels the excess air requirement decreased with increase in load (table 4.19). Similar trend was observed for diesel fuel with lower that of biodiesel. The excess air requirement increased with increase in amount of biodiesel in the blends.

b. Exhaust gas temperature

The exhaust gas temperature gives an indication the amount of waste heat going with exhaust gases. The exhaust gas temperature was also recorded by using KM900 exhaust gas analyzer attached with a k type thermocouple. The different loads were applied on the engine testing and effect of loads on exhaust gas temperature are described as below:

The exhaust gas temperature of the different biodiesel blends is shown in Fig. 4.27. From the figure it was clear that the exhaust gas temperature increased with increase in load and amount of blended biodiesel in the fuel.

At 2 kW load the exhaust gas temperature was observed to be higher in biodiesel fuel mode by 38°C as compared with diesel fuel mode. The exhaust gas temperature varied between 292 to 310°C for jatropha biodiesel blended fuels. The raise in temperature increased with increase in blended amount of biodiesel. The highest exhaust gas temperature was 314°C for jatropha biodiesel whereas for diesel it was 276°C at 50 per cent load on engine.

The exhaust gas temperature of blended fuel and biodiesel at 2.5 kW load increased as compared with 50 per cent load. Maximum exhaust gas temperature at 2.5 kW load conditions was noted as 355°C for biodiesel whereas that of diesel fuel was 338°C. For the jatropha biodiesel blended fuels, the exhaust gas temperature followed the same trend of 2 kW load with a difference in temperature of 4°C. The exhaust gas temperature increased from 314 to 355°C for biodiesel from 2 to 2.5 kW load conditions.

The exhaust gas temperature of blended fuels and biodiesel at 3.5 kW load condition was 19 per cent higher than 2 to 2.5 kW load conditions. The highest exhaust gas temperature was observed as 463°C for biodiesel among three load conditions. The raise in exhaust gas temperature of 3.5 kW load followed similar trend of blended fuels. The diesel fuel mode exhaust gas temperature was observed as 375°C.

The exhaust gas temperature reflects on the status of combustion inside the combustion chamber (Nichaus *et al.*, 1986). The exhaust gas temperature increased with increase in load and amount of biodiesel blends. So the raise in the exhaust gas temperature may be due to ignition delay and increased quantity of fuel

injection. The exhaust gas temperature can be reduced by adjusting the injection timing/injection pressure or injection timing of fuel in to the diesel engine.

c. Carbon dioxide (CO₂) emission

The carbon dioxide emission from the diesel engine with different blends is shown in Fig. 4.28. The CO₂ increased with increase in load conditions for diesel fuel and for biodiesel blended fuel. The jatropha biodiesel followed the same trend of CO₂ emission, which was higher than in the case of diesel fuel. The CO₂ in the exhaust gas was same for jatropha biodiesel blended fuels and jatropha biodiesel. This is good accordance with results obtained by Masjuki *et al.* (1993).

d. Carbon monoxide (CO) emission

KM900 model exhaust gas analyzer was used to monitor the CO emission in exhaust gas from the diesel engine. The CO emission from the diesel fuel with biodiesel blended fuels and biodiesel is shown in Fig. 4.29. It was evident that CO emission decreased with increase of amount of biodiesel blend with diesel for all load conditions. The CO reduction by biodiesel was 16, 14 and 14 per cent at 2, 2.5 and 3.5 kW load conditions. With diesel fuel mode the CO was recorded as lowest (520 ppm) at 2 kW load and as load increased to 3.5, CO also increased to 898 ppm. Similar results were obtained for biodiesel blended fuels and jatropha biodiesel with lower emission than diesel fuel. The amount of CO emission was lower in case of biodiesel blended fuels and biodiesel than diesel fuel because of the fact that biodiesel contained 10 per cent oxygen molecules. This may lead to the reduction CO emission in biodiesel fuelled engine.

e. NO_x emission

The NO_x emission from engine with different jatropha biodiesel blended fuels and biodiesel is shown in Fig. 4.30. From the figure it was concluded that the NO_x emission increased with increase in biodiesel amount in the blended fuels and also found that NO_x emission from the biodiesel fuel was higher than diesel fuel. The NO_x emission increased for biodiesel by 15, 18 and 19 per cent higher than diesel fuel at 2, 2.5 and 3.5 kW load conditions. The percentage of increase in NO_x concentration for blended biodiesel fuels were observed as 6.6 to 19 per cent when compared with diesel fuel. NO_x reductions were in conformity with results reported by Schumacher *et al.* (1993) for soyate methyl ester fuel.

Probable reasons for increase in NO_x concentration by about 2 to 10 per cent from biodiesel fuelled engine was due to higher oxygen level in the fuel and the type of engine according to Anon, (1993). Forgiel and Varde (1981) observed the NO_x concentration depended on the size of orifice. They reported that the NO_x increased when the size orifice was reduced. Heywood (1988) reported that the NO_x formation depended on combustion temperature and availability of oxygen. Fosseen and Goetz (1993) reported that the NO_x concentration can be reduced by advancing the beginning of injection time by 0-3°. However, the increase in NO_x concentration is the main problem in biodiesel and it can be reduced by suitable change in the engine parameters.

f. Oxygen (O₂) emission

The O₂ emission from the diesel engine was reduced with increase in load (Fig. 4.31). The higher O₂ emission was observed at 2 kW load condition. At all tested

load conditions the O₂ emission level was almost same for jatropha biodiesel blended fuel and biodiesel when compared with diesel fuel. The O₂ emission in the exhaust gas at 3.5 kW load was lower than other testing loads. For all tested fuels the lower O₂ emission at 3.5 kW was observed and it may due to more O₂ used in combustion of higher quantity of fuel. This is good agreement with results obtained by Masjuki *et al.* (1993).

4.8. Cost economics

The cost economics for jatropha biodiesel production determined by making suitable assumption are given in the section

Fixed cost

Cost of biodiesel pilot plant	=	Rs. 150000/-
Salvage value	=	Rs. 15000/-
Life period	=	15 years
Depreciation per year	=	Rs. 9,000/-
Depreciation per day	=	Rs. 25/-
Interest per year (12%)	=	Rs. 18000/-
Interest per day	=	Rs. 49.50/-
Housing cost per year	=	Rs. 25000/-
Housing cost per day	=	Rs. 68.50/-
Total fixed cost per day	=	Rs. 143/-

Variable cost

Processing capacity of biodiesel pilot plant	=	250 litres/day
Production of biodiesel per day	=	250 litres
Cost of jatropha oil per litre	=	Rs. 12/-
Cost of feed stock (250 litres) per day	=	Rs. 3000/-

Labour charges per day (2 persons @Rs. 120 per person per day)	=	Rs. 240/-
Electricity charges per day	=	Rs. 60/-
Quantity of water used per day	=	500 litres
Cost of water used per day	=	Rs. 125/-
Cost of catalyst (NaOH) per day	=	Rs. 100/-
Cost of methanol per day	=	Rs. 1500/-
Repair cost per annum	=	Rs. 7500/-
Repair cost per day	=	Rs. 20.55/-
Total variable cost per day	=	Rs. 5045.55/-
Total cost for production of biodiesel per day	=	Rs. 5188.55/-
By product production per day, kg	=	Rs. 37.50/-
Cost of by product per kg	=	Rs. 20/-
Income from the by product per day	=	Rs. 750/-
Cost per day of 250 litres biodiesel production	=	Rs.5188.55/-
Net cost per day of 250 litres biodiesel production	=	Rs. 4438.55/-
Cost of biodiesel per litre	=	Rs. 17.75/-
Cost of biodiesel per litre for sale	=	Rs. 22/-
Total in come by the biodiesel per day	=	Rs. 1062.5/-
Cost benefit ratio	=	4.18

CHAPTER V

SUMMARY AND CONCLUSIONS

The results of the investigations carried out to study the properties of selected oils, biodiesel production, biodiesel properties, performance and emissions of biodiesel fuelled engine are summarised in this chapter.

1. The calorific values for tested oils were ranged from 38.28 to 40.17 MJ/kg. The higher heating values of oils were ranged from 86 to 90 per cent of that of diesel fuel.
2. The kinematic viscosity of pungam, palm, pinnay, jatropha, neem, coconut and eluppai oil at 40°C was 8.6, 11.3, 8.4, 7.5, 7.6, 5.7 and 9.3 times higher than the kinematic viscosity of diesel fuel respectively.
3. The specific gravity of selected tree seed oils ranged from 0.9102 to 0.9261, which was higher as compared with diesel fuel (0.8396). The maximum value of specific gravity was observed for palm oil and minimum for pungam oil.
4. The flash point of the selected tree seed oils were found to be more than 200°C and maximum flash point was observed for neem oil (288°C) and the lowest was recorded for pinnay oil (211°C).
5. The cloud point of the selected tree seed oils ranged from 13 to 18°C. The highest cloud point was observed for eluppai oil (18°C) and the lowest was 13°C for jatropha oil.

6. The lowest pour point was recorded for neem oil and the highest for coconut oil (13°C) whereas the pour point of diesel fuel was observed as 1°C.
7. The carbon residue of selected tree seed oils was observed between 0.45 and 0.64 per cent. The highest carbon residue was obtained for eluppai oil and the lowest carbon residue for coconut oil.
8. The ash content of selected tree seed oils varied from 0.011 to 0.021 per cent. The maximum ash content was obtained for eluppai oil and the minimum for coconut oil.
9. Free Fatty Acids of selected tree seed oils varied from 1.44 to 17.3 per cent. The total glycerol content varied between 12.36 and 15.08 per cent. The highest value was recorded for jatropha oil and the lowest for coconut oil.
10. By heating the oils the viscosity of oils was brought to viscosity of diesel fuel by heating the raw oil above 100°C. In case of blending of oils with diesel, the maximum reduction in viscosity was achieved by B₂₀ raw oil blended fuel having equivalent specific gravity of diesel and 1.7 times higher viscosity than diesel. The fuels obtained by these methods viz., heating the oils and blending of oils with diesel were found to be unsuitable for diesel engines.
11. By using transesterification to produce biodiesel, Methyl ester conversion ranged from 97.98 to 99.47 per cent. The maximum conversion was obtained from the jatropha oil and the lowest for neem oil.
12. Biodiesel production from eluppai and pinnay oils were failed and gel formation was observed due to higher free fatty acid content.

13. The molar ratio of 1:6, catalyst amount of 0.3 M and reaction time of 2 hours were found to be the optimum conditions for pungam biodiesel production.
14. The optimal conditions for best yield of biodiesel from jatropha oil was observed with molar ratio of 1:6, catalyst amount of 0.2 M and reaction time of 2 hours.
15. Material flow in pilot biodiesel plant for jatropha biodiesel production from 15 kg of jatropha oil, 14.9 kg of pure biodiesel and 2.25 kg of glycerol were produced.
16. The heat contents of biodiesels were approximately 10 to 14 per cent lower than that of diesel. The calorific values for produced biodiesels were ranged from 38.11 to 39.31 MJ/kg.
17. The kinematic viscosity of biodiesel from pungam, palm, jatropha, neem and coconut oils at 40°C was found to be 1/7.5, 1/9.3, 1/6.8, 1/6.5 and 1/5.5th of the viscosity of raw oil respectively.
19. After the transesterification, the specific gravity of biodiesel varied from 0.8618 to 0.885 at 40°C whereas the specific gravity of raw oils was observed to be between 0.9581 and 0.9098.
20. The flash points of biodiesel was noted as 3 to 3.5 times higher than the diesel flash point (51°C). The coconut biodiesel recorded the lowest flash point temperature (156°C) and maximum for neem biodiesel (179°C).
21. The highest pour point was observed for neem biodiesel (-1°C), which was 2°C lower than the diesel fuel. The lowest pour point was observed for coconut

biodiesel (11°C). The cloud point of biodiesel was noted as 16 to 14°C and was slightly higher than (1 to 2°C) the raw oils.

22. The observed carbon residue of biodiesels varied between 0.24 and 0.32 per cent. The lower carbon residue was recorded for coconut biodiesel (0.18%), which was 0.35 per cent for raw coconut oil. The ash content of biodiesels varied between 0.010 and 0.015 per cent. The lowest ash content was observed for coconut biodiesel and maximum for palm biodiesel.
23. The free fatty acid of the biodiesels was varied from 0.27 to 1.20 per cent for palm and neem biodiesel. The observed total glycerol content of biodiesels varied between 0.08 to 0.25 per cent. There was no free glycerol observed in palm, jatropha and coconut biodiesels.
24. A biodiesel pilot plant was developed for biodiesel production from tree seed oils. The plant used for jatropha biodiesel production, from 15 kg of jatropha oil, 14.9 kg of pure biodiesel was obtained.
25. In jatropha biodiesel blended fuels, increase in amount of diesel fuel showed no effect on specific gravity of fuels. The viscosities of biodiesel-blended fuel have little effect on reducing the viscosity of biodiesel. The calorific value of blended fuels was improved by addition of diesel fuel in the biodiesel blends. The flash point of blended fuels was increased with increase in amount of biodiesel in the fuels.
26. A 5.2 kW diesel engine with alternator was used to test *Jatropha curcas* biodiesel and its blends. In the case of jatropha biodiesel alone, the fuel consumption in the diesel engine was about 14 per cent higher than that of

diesel. The per cent increase in specific fuel consumption ranged from 3 to 14 for B₂₀ to B₁₀₀ fuels. The brake thermal efficiency for biodiesel and its blends was found to be slightly higher than that of diesel fuel at tested load conditions and there was no difference between the biodiesel and its blended fuels efficiencies.

27. For jatropha biodiesel and its blended fuels, the exhaust gas temperature increased with increase in load and amount of biodiesel. The highest exhaust gas temperature was observed as 463°C for biodiesel among the three load conditions. The diesel mode exhaust gas temperature was observed as 375°C.
28. The CO₂ emission from the biodiesel fuelled engine was slightly higher than diesel fuel as compared with diesel. The carbon monoxide reduction by biodiesel was 16, 14 and 14 per cent at 2, 2.5 and 3.5 kW load conditions.
29. The NO_x emission from biodiesel increased by 15, 18 and 19 per cent higher than that of the diesel fuel at 2, 2.5 and 3.5 kW load conditions.
30. The cost of jatropha biodiesel per litre was worked out as Rs 22/-

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APPENDICES

A1. Effect of heating of raw oils on kinematic viscosity

	Pungam	Palm	Pinnay	Jatropha	Neem	Coconut	Eluppai
100	17.57	17.80	13.22	16.50	18.70	15.15	22.13
90	18.38	19.40	18.11	19.20	21.27	17.64	24.33
80	20.33	23.70	20.83	23.60	23.27	19.21	26.14
70	22.16	27.60	24.78	28.10	26.64	20.36	29.39
60	29.62	30.00	29.36	31.20	32.86	22.36	34.52
50	34.83	32.67	34.60	34.05	34.71	24.33	39.40
40	41.60	54.74	40.42	36.31	36.75	27.75	45.35

A2. Kinematic viscosity of raw oil blended fuels

	Pungam	Palm	Pinnay	Jatropha	Neem	Coconut	Eluppai
B ₁₀₀	41.58	54.70	40.37	36.25	36.89	27.48	45.32
B ₈₀	24.62	44.01	26.90	21.73	25.31	18.08	27.50
B ₆₀	16.51	34.73	15.42	14.11	18.91	11.27	19.88
B ₄₀	10.99	16.12	10.79	10.16	12.14	9.99	12.98
B ₂₀	8.71	8.70	8.65	8.62	8.39	7.79	8.59

A3. Methyl ester conversion for pungam biodiesel in bench scale reactor

Biodiesel source	Molar ratio	Catalyst amount, M	Methyl ester conversion, %			
			I	II	III	Average
Pungam	1:4.5	0.1	75.70	75.87	75.93	75.80
		0.2	79.80	79.30	79.90	79.70
		0.3	82.20	82.33	82.47	82.30
	1:6	0.1	86.52	86.70	86.48	86.60
		0.2	89.20	89.35	89.45	89.30
		0.3	91.20	91.22	91.58	91.30
	1:7.5	0.1	94.58	94.60	94.82	94.65
		0.2	96.80	96.85	96.75	96.80
		0.3	98.70	98.45	98.75	98.65

A4. Methyl ester conversion for palm biodiesel in bench scale reactor

Biodiesel source	Molar ratio	Catalyst amount, M	Methyl ester conversion, %			
			I	II	III	Average
Palm	1:6	0.1	92.70	92.68	92.72	92.70
		0.2	93.40	93.45	93.55	93.45
		0.3	94.65	94.70	94.75	94.70
	1:7.5	0.1	98.63	98.90	98.77	96.90
		0.2	99.20	99.08	99.12	99.15
		0.3	99.47	99.50	99.73	99.70
	1:9	0.1	99.70	99.75	99.85	97.30
		0.2	99.60	99.82	99.78	99.55
		0.3	99.40	99.54	99.66	99.50

A5. Methyl ester conversion for jatropha biodiesel in bench scale reactor

Biodiesel source	Molar ratio	Catalyst amount, M	Methyl ester conversion, %			
			I	II	III	Average
Jatropha	1:4.5	0.1	95.10	95.35	95.45	95.10
		0.2	96.28	96.10	96.32	97.50
		0.3	97.30	97.43	97.57	99.20
	1:6	0.1	97.50	97.45	97.55	96.30
		0.2	98.38	98.10	98.62	98.50
		0.3	99.70	99.66	99.74	99.70
	1:7.5	0.1	99.20	99.10	99.30	99.20
		0.2	99.70	99.50	99.30	99.50
		0.3	99.70	99.75	99.65	99.70

A6. Methyl ester conversion for neem biodiesel in bench scale reactor

Biodiesel source	Molar ratio	Catalyst amount, M	Methyl ester conversion, %			
			I	II	III	Average
Neem	1:6	0.1	82.70	82.65	82.55	82.65
		0.2	86.73	86.60	86.67	86.65
		0.3	89.48	89.30	89.72	89.45
	1:7.5	0.1	91.10	91.23	91.27	91.20
		0.2	93.70	93.72	93.88	93.75
		0.3	94.75	94.60	94.85	94.70
	1:9	0.1	96.30	96.54	96.86	96.50
		0.2	97.60	97.67	97.53	97.60
		0.3	98.65	98.50	98.75	98.60

A7. Methyl ester conversion for coconut biodiesel in bench scale reactor

Biodiesel source	Molar ratio	Catalyst amount, M	Methyl ester conversion, %			
			I	II	III	Average
Coconut	1:6	0.1	92.28	92.30	92.32	92.30
		0.2	93.90	93.87	93.93	93.90
		0.3	94.85	94.75	94.90	94.80
	1:7.5	0.1	99.14	99.14	99.10	99.12
		0.2	99.82	99.89	99.89	99.80
		0.3	99.52	99.56	99.60	99.56
	1:9	0.1	99.03	99.02	99.09	99.04
		0.2	99.35	99.37	99.39	99.37
		0.3	99.66	99.72	99.74	99.71

A8. Methyl ester conversion for pungam biodiesel in bench scale reactor

Biodiesel source	Molar ratio	Catalyst amount, M	Reaction time, h	Methyl ester conversion, %		
				I	II	Average
Pungam	1:5.25	0.2	0.5	86.40	86.40	86.40
			1.0	89.26	89.21	89.23
			1.5	92.14	92.25	92.19
			2.0	95.47	95.41	95.44
	1:5.25	0.3	0.5	87.12	87.27	87.19
			1.0	92.40	92.50	92.45
			1.5	96.24	96.12	96.33
			2.0	97.60	97.42	97.51
	1: 6	0.2	0.5	89.14	89.54	89.34
			1.0	94.60	94.70	94.65
			1.5	96.40	96.57	95.48
			2.0	97.48	98.14	97.81
	1: 6	0.3	0.5	92.40	92.60	92.50
			1.0	94.50	95.70	95.10
			1.5	96.74	96.76	96.74
			2.0	98.30	98.40	98.35

A8. Methyl ester conversion for jatropha biodiesel in bench scale reactor

Biodiesel source	Molar ratio	Catalyst amount, M	Reaction time, h	Methyl ester conversion, %		
				I	II	Average
Jatropha	1: 4.5	0.1	0.5	65.50	65.62	65.56
			1.0	72.50	72.54	72.52
			1.5	77.25	77.33	77.29
			2.0	78.80	78.70	78.75
	1: 4.5	0.2	0.5	78.03	78.01	78.02
			1.0	79.34	79.62	79.48
			1.5	80.92	80.98	80.95
			2.0	81.27	81.37	81.32
	1: 4.5	0.3	0.5	75.79	78.85	75.82
			1.0	77.23	77.35	77.29
			1.5	79.52	79.44	79.48
			2.0	80.14	80.30	80.22
	1:5.25	0.1	0.5	82.67	82.73	82.70
			1.0	84.85	84.95	84.90
			1.5	86.80	87.20	87.00
			2.0	87.72	87.68	87.70
	1:5.25	0.2	0.5	84.36	84.42	84.39
			1.0	86.33	86.37	86.35
			1.5	88.14	88.46	88.30
			2.0	89.58	89.62	89.60
	1:5.25	0.3	0.5	86.30	86.24	86.27
			1.0	87.80	87.90	87.85
			1.5	89.24	89.16	89.20
			2.0	89.47	90.53	90.00
	1: 6	0.1	0.5	84.03	83.97	84.00
			1.0	89.28	89.32	89.30
			1.5	92.35	92.43	92.39
			2.0	96.65	96.83	96.74
	1: 6	0.2	0.5	85.06	85.28	85.17
			1.0	90.55	90.65	90.60
			1.5	97.43	97.47	97.45
			2.0	99.12	99.10	99.11
	1: 6	0.3	0.5	88.27	88.51	88.39
			1.0	93.72	93.80	93.76
			1.5	98.04	98.08	98.06
			2.0	99.12	99.27	99.20

Table 2.4. Comparison of different national standards for biodiesel (LAAF, 1999)

	Austria	France	Germany	Italy	Sweden	USA
Standard /Specification	ON C1191	Journal Official	DIN V 51606	UNI 10635	SS 155436	ASTM D-6751
Date of issue	July 1997	Sep 1997	Sep 1997	April 1997	Nov 1996	July 1999
Application	Fatty Acid Methyl Ester	Vegetable Oil Methyl Ester	Fatty Acid Methyl Ester	Vegetable Oil Methyl Ester	Vegetable Oil Methyl Ester	Fatty Acid Mono Alkyl Ester
Density 15°C, g/cm	0.85 - 0.89	0.87 - 0.90	0.875 - 0.90	0.86 -0.90	0.87 - 0.90	-
Viscosity at 40°C, mm ² /s	3.5-5.0	3.5-5.0	3.5-5.0	3.5-5.0	3.5-5.0	1.9-6.0
Distillation, 95%, °C	-	<360	-	<360	-	-
Flashpoint °C	>100	>100	>110	>100	>100	130°C minimum
Pour point, °C	-	<-10	-	<0/<-15	-	-
Sulfur, % mass	<0.02	-	<0.01	<0.01	<0.001	<0.05
CCR 100%, % mass	<0.05	-	<0.05	-	-	<0.05
Ash % mass	-	-	-	<0.01	<0.01	-
Cetane Number	>49	>49	>49	-	>48	>47
Ester content, % mass	-	>96.5	-	>98	>98	-
Triglyceride, % mass	-	<0.2	<0.4	<0.1	<0.1	-
Free glycerol, % mass	<0.02	<0.02	<0.02	<0.05	<0.02	<0.02
Total glycerol % mass	<0.24	<0.25	<0.25	-	-	<0.24
Alkalinity, mg/kg	-	<5	<5	-	<10	-

Table 2.5. Indian requirement for diesel fuels (IS: 1460-2000)

S. No.	Characteristics	Requirement
1.	Acidity	Nil
2.	Inorganic acidity, total, mg KOH/g (maximum)	0.50
3.	Ash, % by mass (maximum)	0.01
4.	Carbon residue (Ramsbottom) on 10 per cent, % by mass (maximum)	0.30
5.	Cetane number (minimum)	42.00
6.	Pour point, °C (maximum)	3.00
7.	Distillation-recovery at 370°C, % by volume (minimum)	95.00
8.	Flash point (Pensky Martens), °C	66.00
9.	Minimum kinematic viscosity at 40°C, cSt	2.0-5.0
10.	Sediment, % by mass (maximum)	0.05
11.	Total sulphur, % by weight (maximum)	1.00

Table 2.3. Experimental conditions for alkaline catalyst based biodiesel production

Sl. No	Oil source	Name of the alcohol used	Catalyst amount used, (%)	Alcohol to oil ratio	Reaction temperature °C	Reaction time, h	Ester conversion, %	References
1	Sunflower	Methanol	MeOH (0.5)	6:1	60-63	1	93-98	Freedman <i>et al.</i> 1984
2	Sunflower	Ethanol	MeOH (0.5)	3:1	<60	1	81	
3	Sunflower	Methanol	MeOH (0.5)	3:1	<75	1	82	
4	Sunflower	Butanol	MeOH (0.5)	3:1	<114	1	88	
5	Triacylglycerol	Methanol	Sodium methanolate (0.2)	7.8:1	-	0.5	97	Lepper and Friesenhagen, 1986
6	Beef tallow	Methanol	NaOH	6:1	60,70-75	2	-	Yusulf Ali and Hanna, 1994
7	<i>Jatropha curcas</i>	Methanol	KOH (0.53M)	4.5:1	30	11	92	Foidl <i>et al.</i> 1996
8	<i>Jatropha curcas</i>	Ethanol	KOH (0.53M)	6.05:1	75	1.5	88.4	
9	Beef tallow	Methanol	NaOH	2-10:1	60	2	80	Praveen <i>et al.</i> 1996
11	Beef tallow	Methanol	NaOH	2-10:1	60	1.5	98	
12	Beef tallow	Methanol	NaOH	2-10:1	60	1.5	90.5	

Table 3.1. Oil bearing trees and their oil content, availability in India

Common name	Botanical name	Oil content, %		Oil seed yield/ha	Available potential (million tonnes)	References
		Whole seed/fruit	Kernel			
Pungam	<i>Pongamia glabra</i>	27	27- 30	1.1 to 29 tonnes	200 (seeds)	Srivastava and Prasad, 2000; Srinivasan, 2003
Palm	<i>Elaeis guineensis</i> Jaq	83	17-21	4 – 6 tonnes of oil	-	Iyer, 1991; Rethinam, 1993
Pinnay	<i>Calophyllum inophyllum</i>	60	70	-	4 (seeds)	Banerji <i>et al.</i> 1983; Bringi, 1987
Jatropha	<i>Jatropha curcas</i> .L	30-40	45-55	0.4 to 10 tonnes	-	Kandapal and Mira, 1995; Vadivel, 2003
Neem	<i>Azadirachta indica</i>	20	45 - 50		500 (seeds)	Srivastava and Prasad, 2000
Coconut	<i>Cocos nucifera</i>	-	63 - 70	6982 nuts	12, 252 (nuts)	Rethinam, 1993; Singh, 2002
Madhuca (Eluppai)	<i>Bassia latifolia</i>	33-43	50	-	0.32 (seeds)	Singh and Sukla, 1991

Table 4.1. Fuel and chemical properties of selected tree seed oils

Property	Diesel	Name of the tree seed oils						
		Pungam	Palm	Pinnay	Jatropha	Neem	Coconut	Eluppai
Fuel properties								
Calorific value, MJ/kg	44.420	40.002	39.787	39.952	39.725	38.279	39.294	40.170
Kinematic viscosity at 40°C, cSt	4.86	41.89	54.70	40.87	36.25	36.75	27.48	45.32
Specific gravity	0.8396	0.9102	0.9261	0.9167	0.9098	0.9247	0.9181	0.9104
Flash point, °C	54	228	248	211	251	288	225	235
Cloud point, °C	13	15	16	15	13	14	17	18
Pour point, °C	1	2	7	6	4	1	13	11
Carbon residue, %	0.21	0.51	0.57	0.48	0.54	0.52	0.45	0.64
Ash content, %	0.01	0.017	0.018	0.015	0.013	0.019	0.011	0.021
Chemical properties								
Free Fatty Acids, %	NA	4.40	1.82	9.30	3.01	5.60	1.44	17.30
Acid value	-	8.756	3.622	18.507	5.97	11.144	2.965	34.477
Total glycerol, %	NA	12.60	13.81	14.43	15.03	12.36	12.60	14.30
Free glycerol, %	NA	0.66	0	1.66	1.579	0.59	0	1.323
Combined glycerol, %	NA	11.94	13.81	12.77	13.451	11.77	12.60	12.98

Table 4.2. Temperature (x) vs kinematic viscosity (y) correlation equations for selected oils

Sl. No.	Name of oil	Correlation equations	R² value	Rate of oil viscosity at 100°C to viscosity of diesel fuel at 40°C	Predicted oil temperature (°C) to match diesel fuel viscosity at 40°C
1	Pungam	$y = -0.4156x + 55.789$	0.9880	3.62	122.5
2	Pinnay	$y = -0.4397x + 56.68$	0.9870	2.72	120.0
3	Palm	$y = -1.033x + 127.500$	0.9897	5.00	119.0
4	Jatropha	$y = -0.344x + 51.052$	0.9880	3.40	118.0
5	Neem	$y = -0.3236x + 50.398$	0.9752	3.85	140.5
6	Coconut	$y = -0.194x + 34.554$	0.9752	3.11	149.5
7	Eluppai	$y = -0.386x + 58.627$	0.9596	4.55	140.0

Table 4.5. Fuel properties of raw and pure biodiesel from seed oils

Oil used	Properties of raw biodiesel				Properties of pure biodiesel				Ratio of water used
	Specific gravity at 40°C	Kinematic viscosity at 40°C, cSt	Sodium content, ppm	pH	Specific gravity at 40°C	Kinematic viscosity at 40°C, cSt	Sodium content, ppm	pH	
Pungam	0.8743	5.71	110	8.7	0.8714	5.59	1.4	7.0	3:1
Palm	0.8861	5.97	115	9.6	0.8850	5.89	1.2	7.0	3:1
Jatropha	0.8630	5.36	9.40	8.5	0.8618	5.32	2.3	7.0	3:1
Neem	0.8784	5.74	42.8	8.7	0.8739	5.68	2.0	7.0	3:1
Coconut	0.8781	5.04	45	8.2	0.8760	4.98	2.2	7.0	3:1

Table 4.7. Fuel and chemical properties of pure biodiesels

Property	Name of the selected biodiesel					Diesel	ASTM Biodiesel standards	DIN 51606 Biodiesel standards
	Pungam	Palm	Jatropha	Neem	Coconut			
Fuel properties								
Calorific value, MJ/kg	39.812	39.518	39.170	38.109	38.985	44.420		
Kinematic viscosity at 40°C, cSt	5.59	5.89	5.32	5.68	4.98	4.86	1.9-6.0	3.5-5.0
Specific gravity	0.8714	0.8850	0.8618	0.8739	0.8760	0.8396	-	0.875-0.890
Flash point, °C	163	164	174	179	156	51	130°C min.	>110
Cloud point, °C	14	14	12	14	16	13	-	-
Pour point, °C	1	5	3	-1	11	1	-	-
Carbon residue, %	0.25	0.30	0.24	0.32	0.28	0.21	<0.50	<0.5
Ash content	0.012	0.015	0.011	0.014	0.010	0.010	-	-
Chemical properties								
Free Fatty Acids, %	1.1	0.27	0.90	1.2	0.975	-	-	
Acid value	2.189	0.537	1.791	2.87	0.925	-	-	<5
Total glycerol, %	0.12	0.17	0.08	0.25	0.08	-	<0.24	<0.25
Free glycerol, %	0.083	0	0	0.18	0	-	<0.02	<0.02
Combined glycerol, %	0.037	0.17	0.08	0.07	0.08	-	-	-

Table 4.19. The performance of diesel engine with different fuels and loads

Sl. No.	Fuel type	Speed, rpm			Brake thermal efficiency, %			Excess air requirement, %		
		2 kW	2.5 kW	3.5 kW	2 kW	2.5 kW	3.5kW	2 kW	2.5 kW	3.5kW
1	B ₀	1526	1504	1494	28.64	30.29	33.01	178.6	137.5	66.5
2	B ₂₀	1524	1502	1494	28.71	30.27	33.07	186.3	145.8	67.3
3	B ₄₀	1524	1502	1494	28.69	30.34	33.10	185.4	147.2	67.9
4	B ₆₀	1524	1502	1492	28.72	30.31	33.04	185.4	147.3	68.7
5	B ₈₀	1522	1504	1490	28.79	30.43	33.11	186.3	148.8	69.1
6	B ₁₀₀	1524	1502	1492	28.84	30.58	33.12	187.3	148.8	71.3

Table 4.20. The alternator output and power output of the engine with tested fuels

Sl. No.	Fuel type	Alternator output						Power output of the engine, hp		
		Voltage, v			Amphere, A					
		2 kW	2.5 kW	3.5 kW	2 kW	2.5 kW	3.5kW	2 kW	2.5 kW	3.5kW
1	B ₀	226	230	223	9.0	11.0	16.0	3.41	4.24	5.95
2	B ₂₀	228	234	226	9.0	11.0	16.0	3.44	4.31	5.98
3	B ₄₀	225	228	230	9.0	11.0	15.5	3.39	4.20	5.98
4	B ₆₀	230	229	222	9.0	11.0	16.0	3.47	4.22	5.95
5	B ₈₀	226	231	223	9.0	11.0	16.0	3.41	4.26	5.98
6	B ₁₀₀	228	233	224	9.0	11.0	16.0	3.44	4.29	5.99

Table 2.2. Oil bearing seeds from trees and their oil content

Common /Botanical name	Oil source	Oil content, %	References
<i>Calophyllum inophyllum</i>	Seed	50	Banerjee <i>et al.</i> 1983
<i>p. saman</i>	Seed	17	
<i>Holoptela integrifolia</i>	Seed	50	
<i>Salvadora oleoides</i>	-	40	
<i>Madhuca butyracea</i>	Seed	17 -19	
<i>M. latifolia</i>	Seed	50	
<i>Moringa oleifera</i>	Seed	27	
Karanj	Kernel	27 –37	Bringi, 1987
Palm	Fruit	65	Singh and Sukla, 1991
Palm	Kernel	52	
Rubber	Whole seed	25	
Salseed	Seed	12	
Khankan	Seed	33	
Palm	Fruit	65	
Palm	Kernel	52	
Jatropha curcas	Seed	35 - 40	Banerjee <i>et al.</i> 1983; Rethinam, 1993
Mahua	Kernel	35-50	Singh and Sukla, 1991; Rethinam, 1993
Neem	Seed	45 - 50	
Coconut	Kernel	67.5	Rethinam, 1993
Cashew	Kernel	35 - 50	
Cashew	Shell	25 - 30	
Almond	Kernel	38 –55	
Tucuma	Pulp	35 – 38	
Tucuma	Kernel	45 - 52	

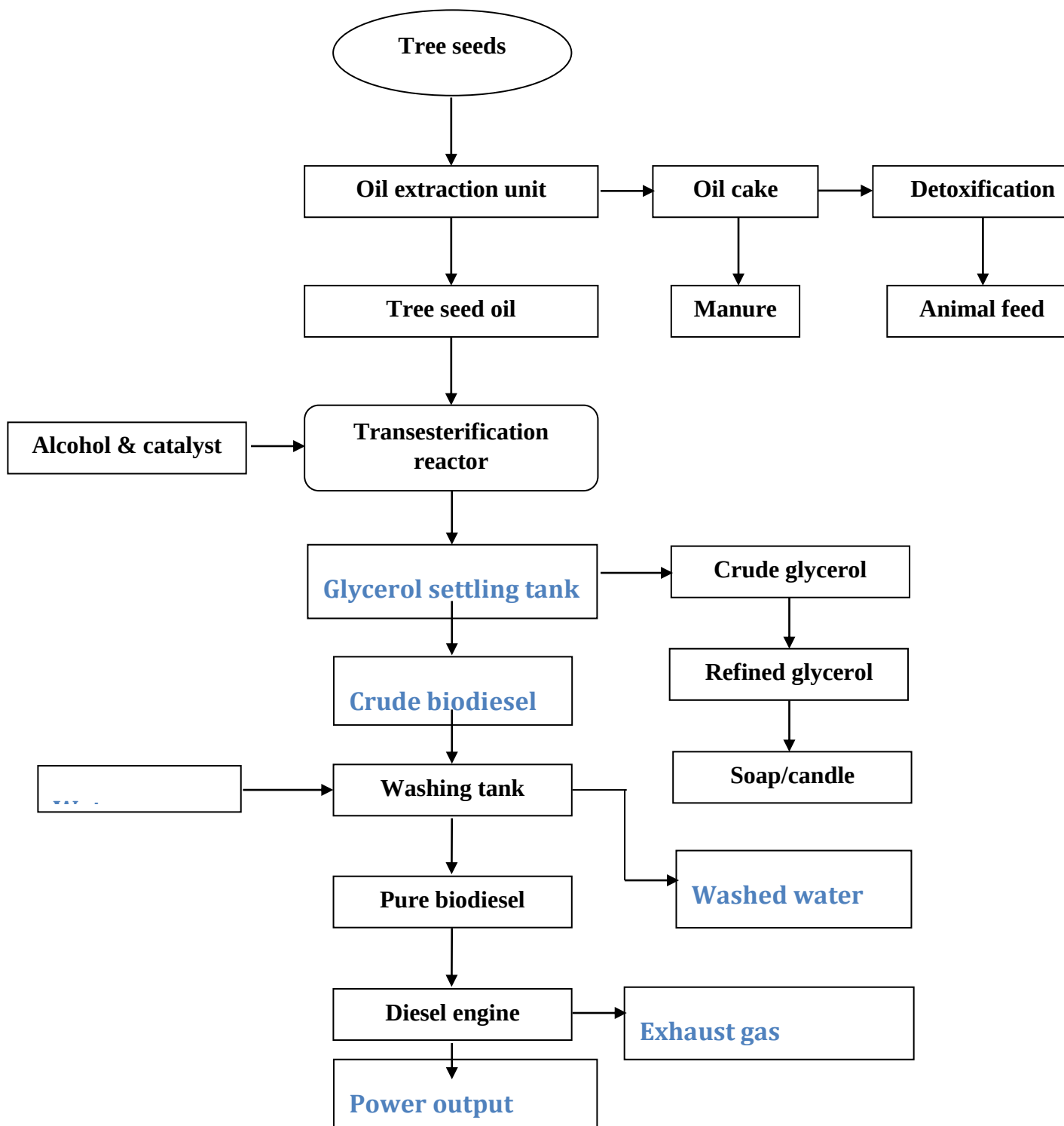


Fig. 3.3. Process flowchart for biodiesel and by product production from tree seed oils

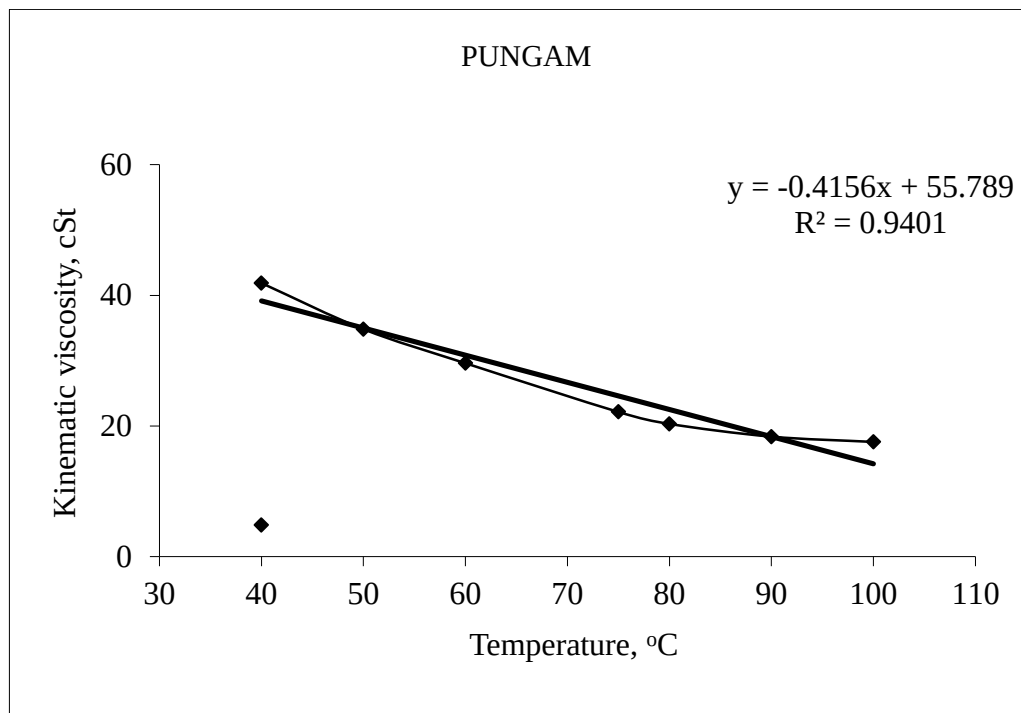


Fig. 4.1. Effect of heating on kinematic viscosity of pungam oil

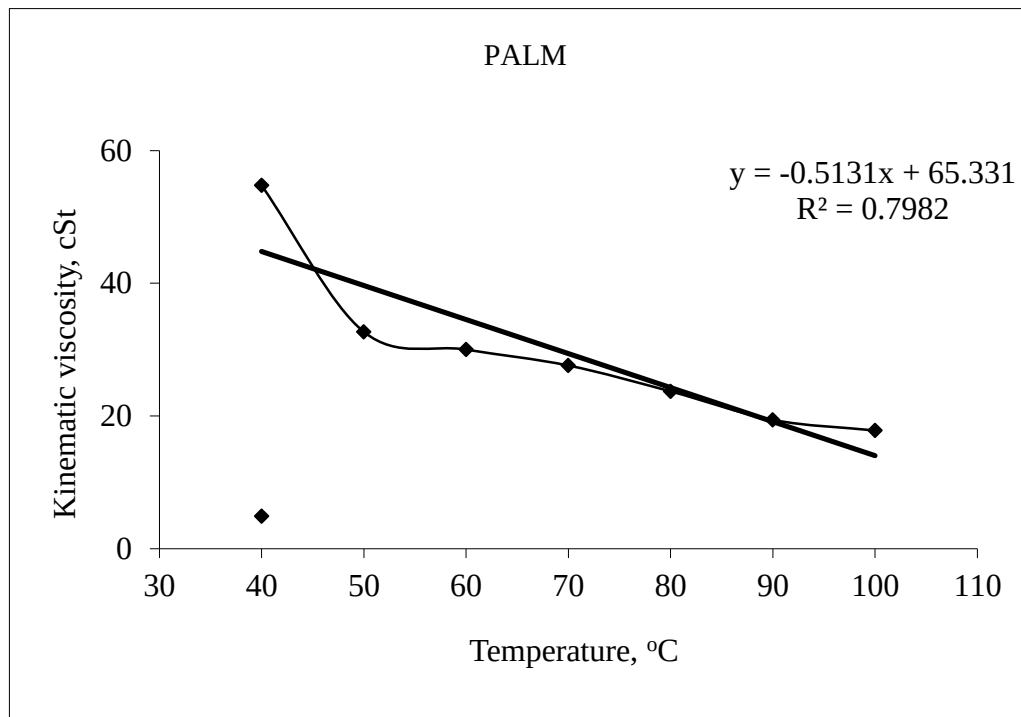


Fig. 4.2. Effect of heating on kinematic viscosity of palm oil

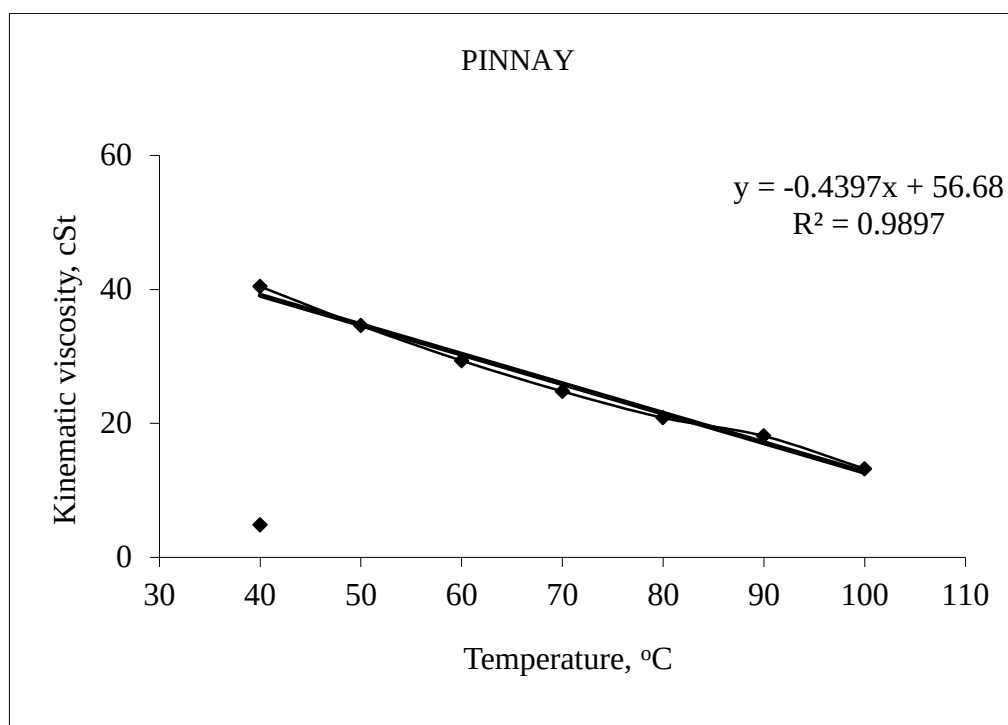


Fig. 4.3. Effect of heating on kinematic viscosity of pinnay oil

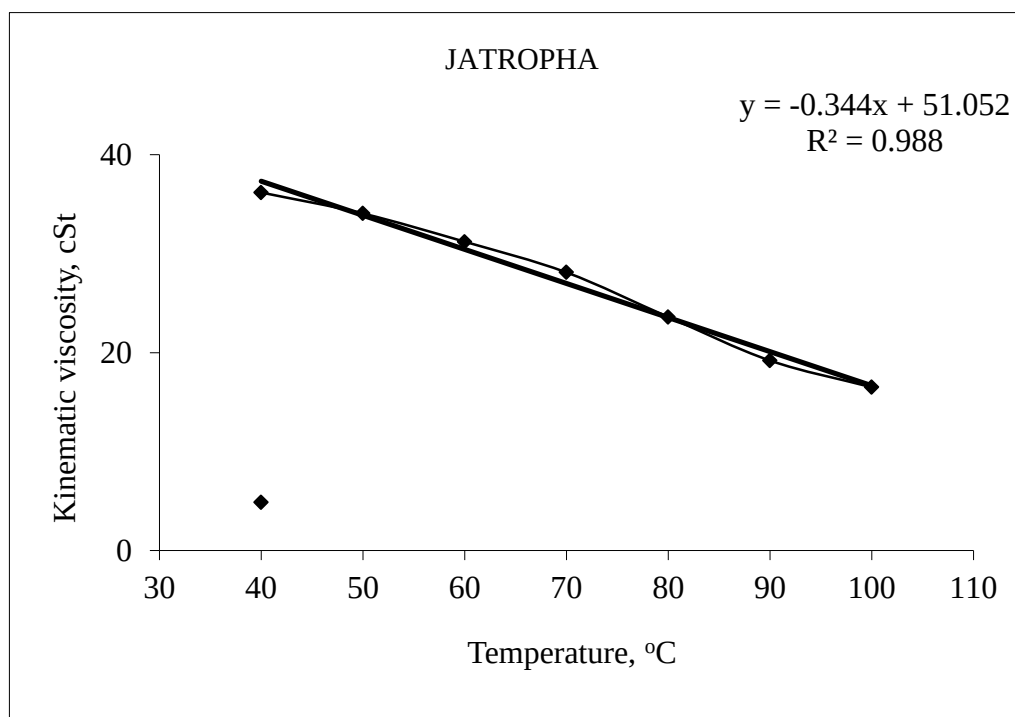


Fig. 4.4. Effect of heating on kinematic viscosity of jatropha oil

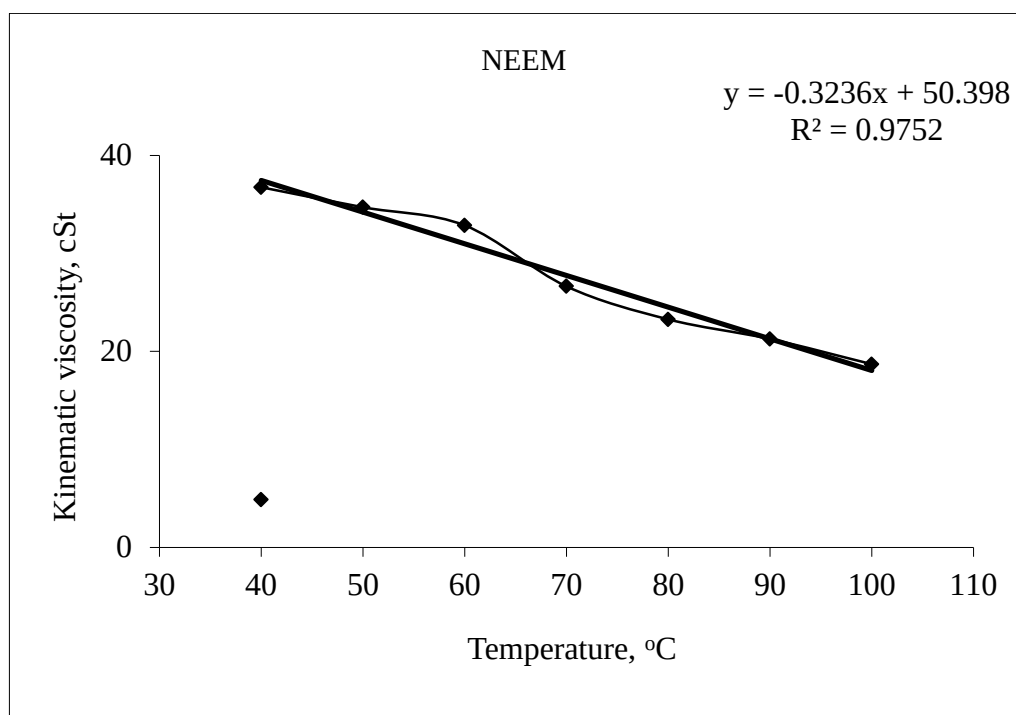


Fig. 4.5. Effect of heating on kinematic viscosity of neem oil

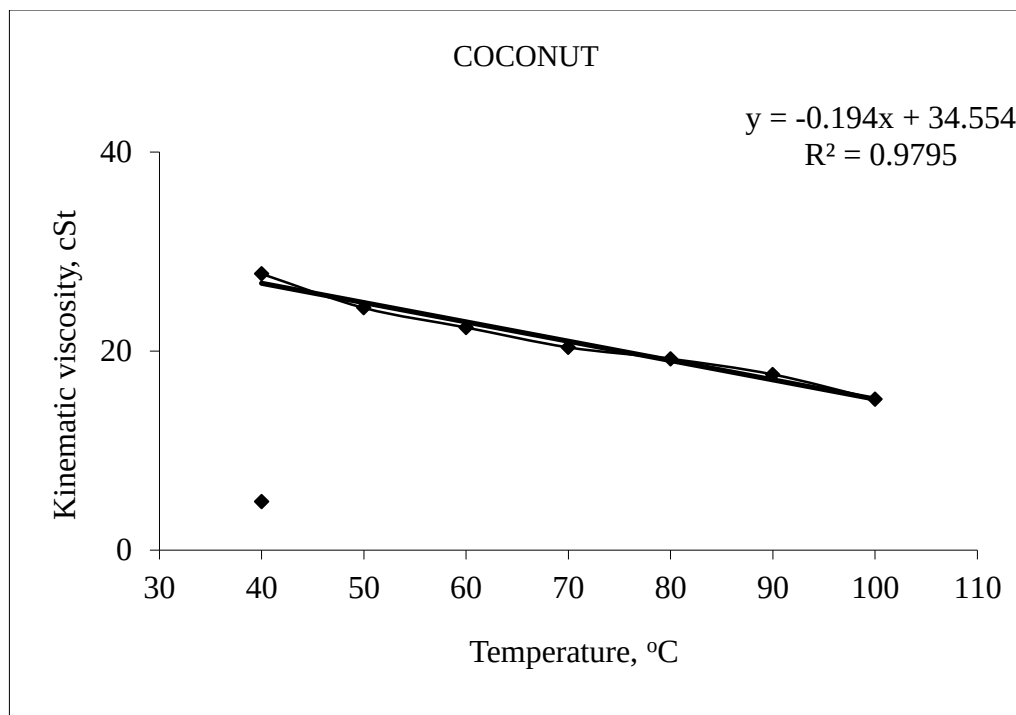


Fig. 4.6. Effect of heating on kinematic viscosity of coconut oil

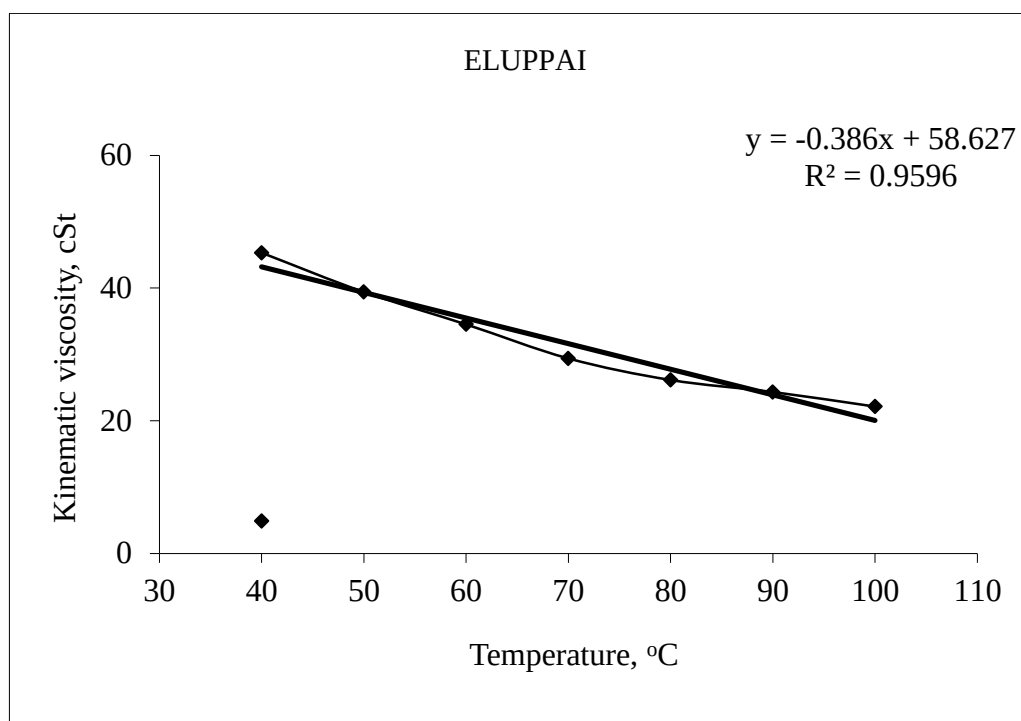


Fig. 4.7. Effect of heating on kinematic viscosity of eluppai oil

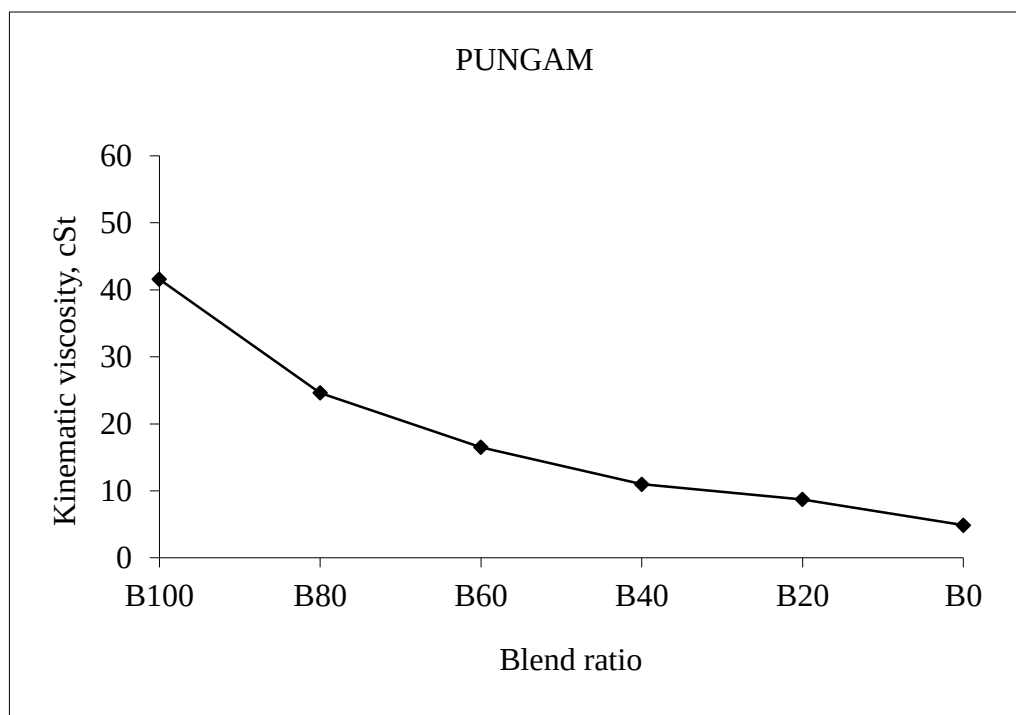


Fig. 4.8. Effect of blending on kinematic viscosity of pungam oil at 40°C

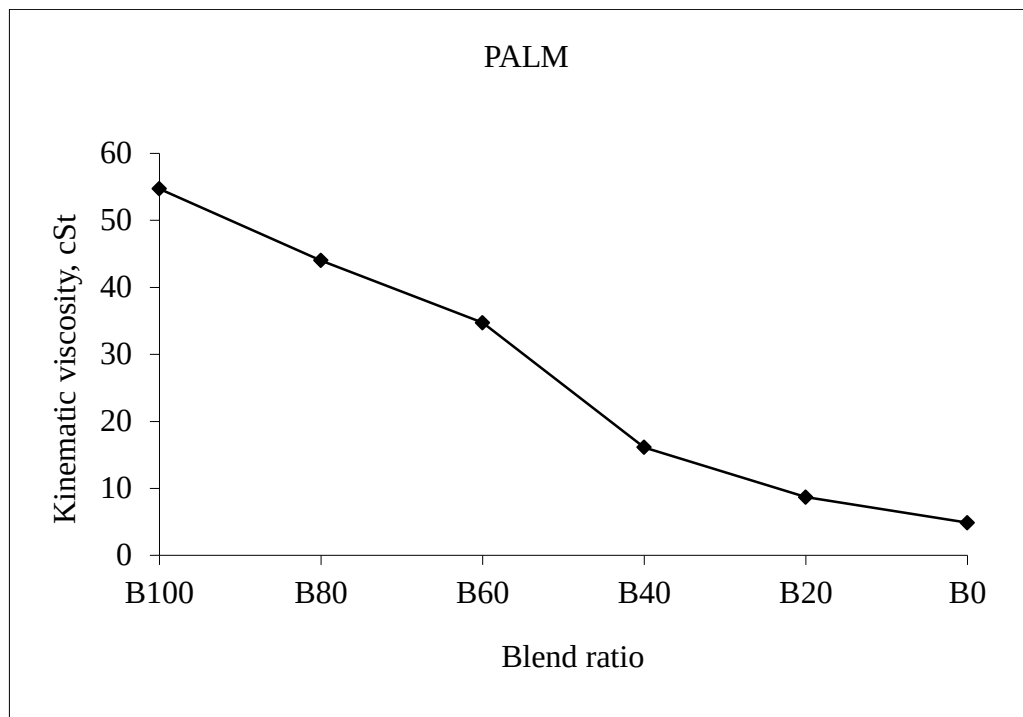


Fig. 4.9. Effect of blending on kinematic viscosity of palm oil at 40°C

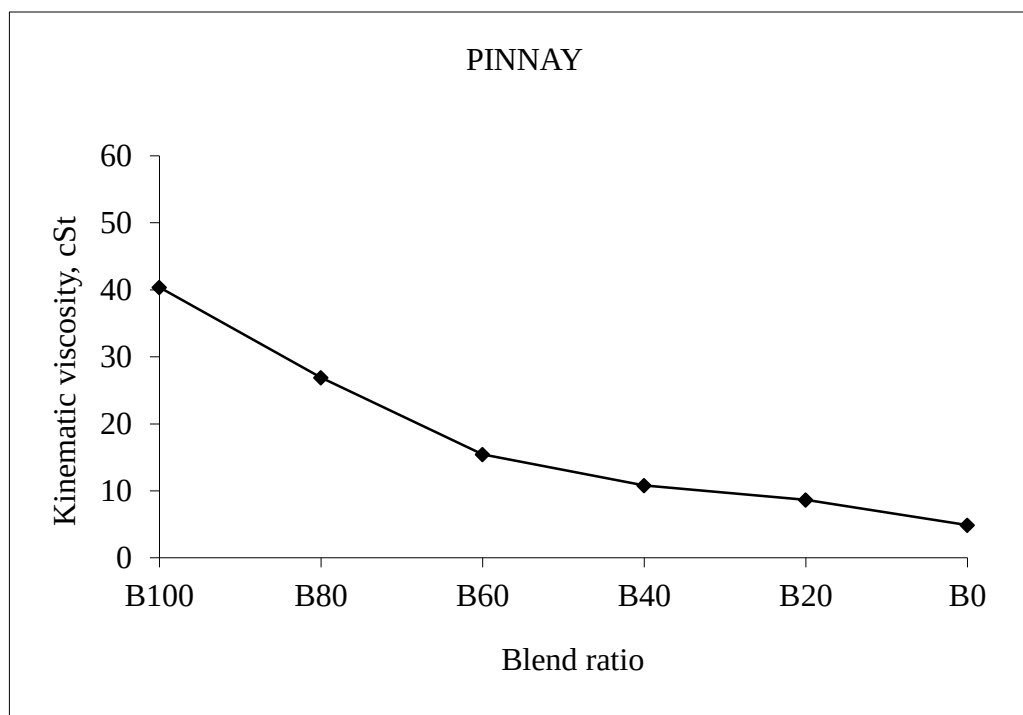


Fig. 4.10. Effect of blending on kinematic viscosity of pinnao oil at 40°C

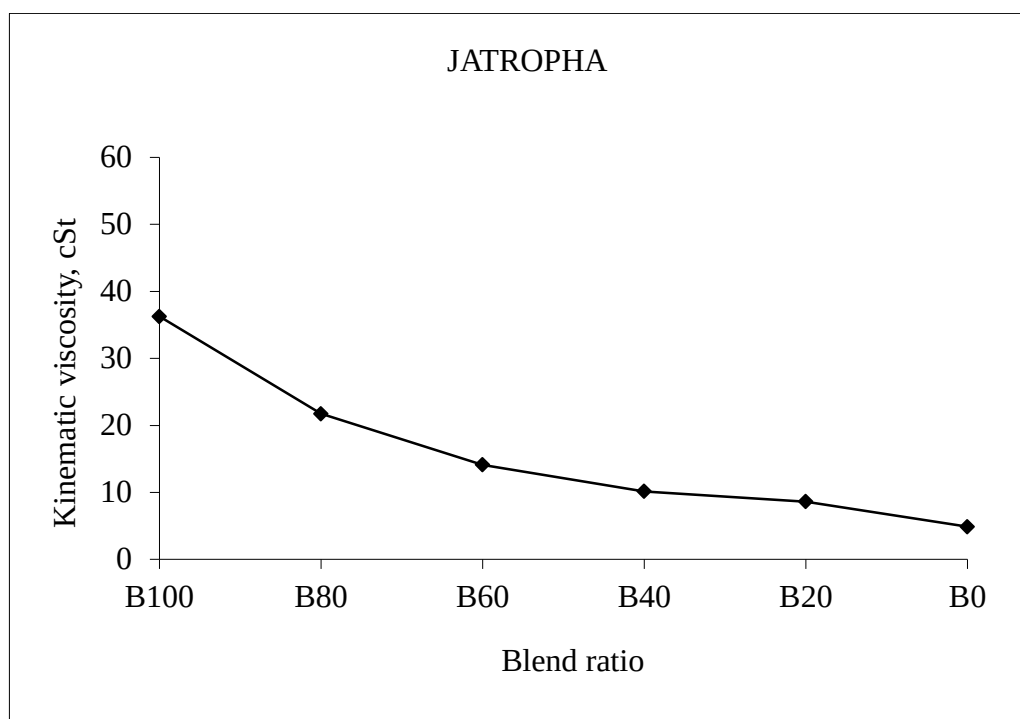


Fig. 4.11. Effect of blending on kinematic viscosity of jatropha oil at 40°C

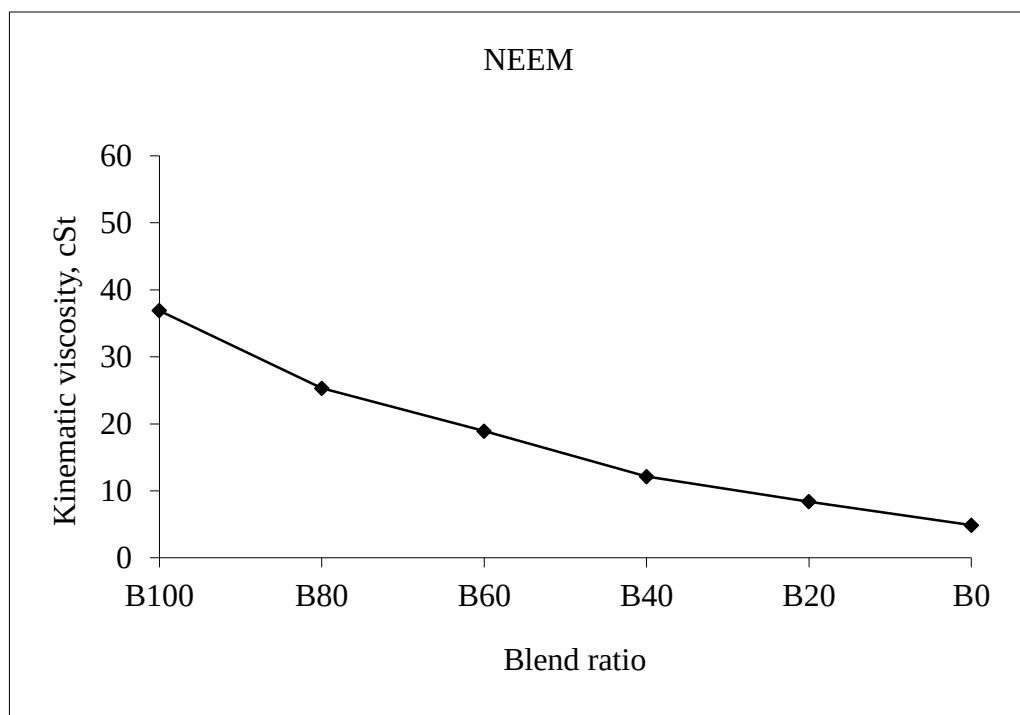


Fig. 4.12. Effect of blending on kinematic viscosity of neem oil at 40°C

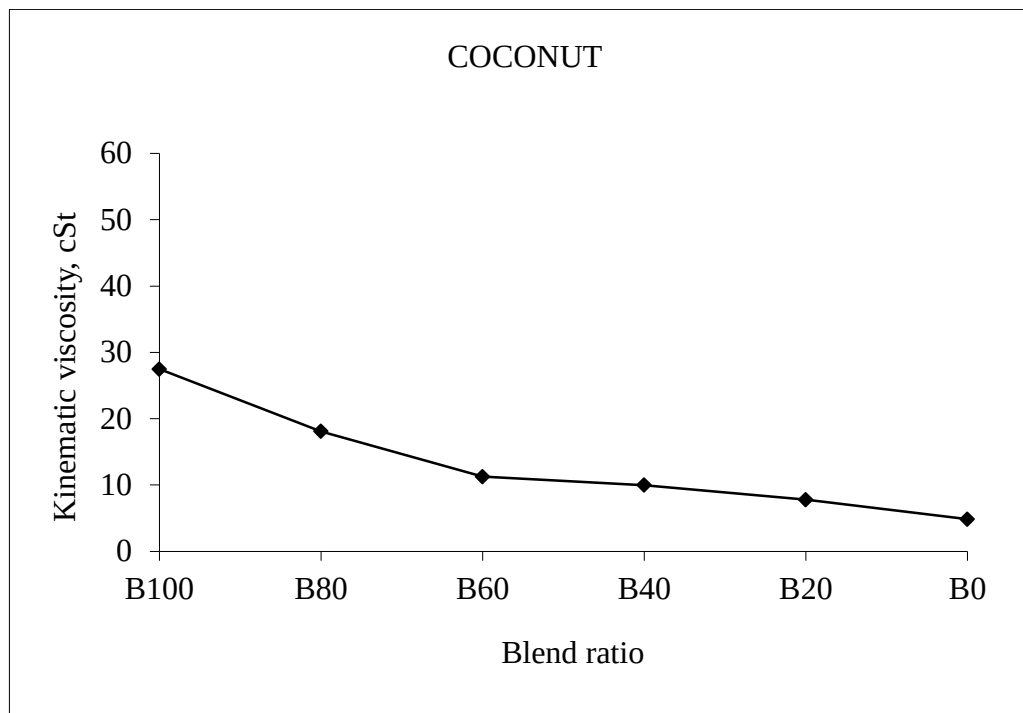


Fig. 4.13. Effect of blending on kinematic viscosity of coconut oil at 40°C

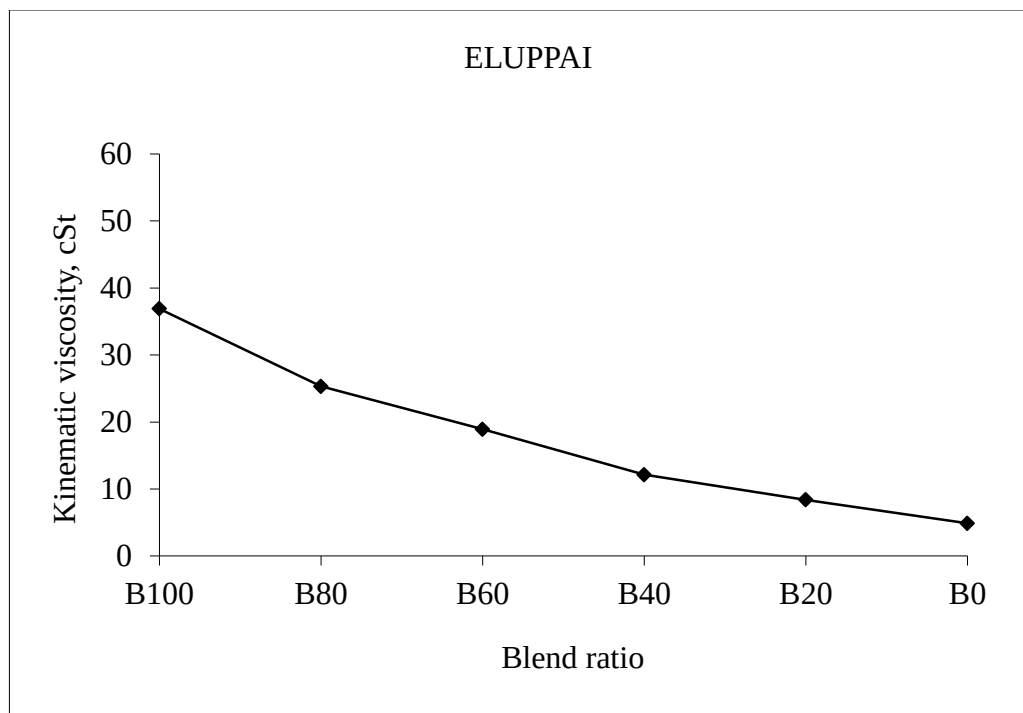


Fig. 4.14. Effect of blending on kinematic viscosity of eluppai oil at 40°C

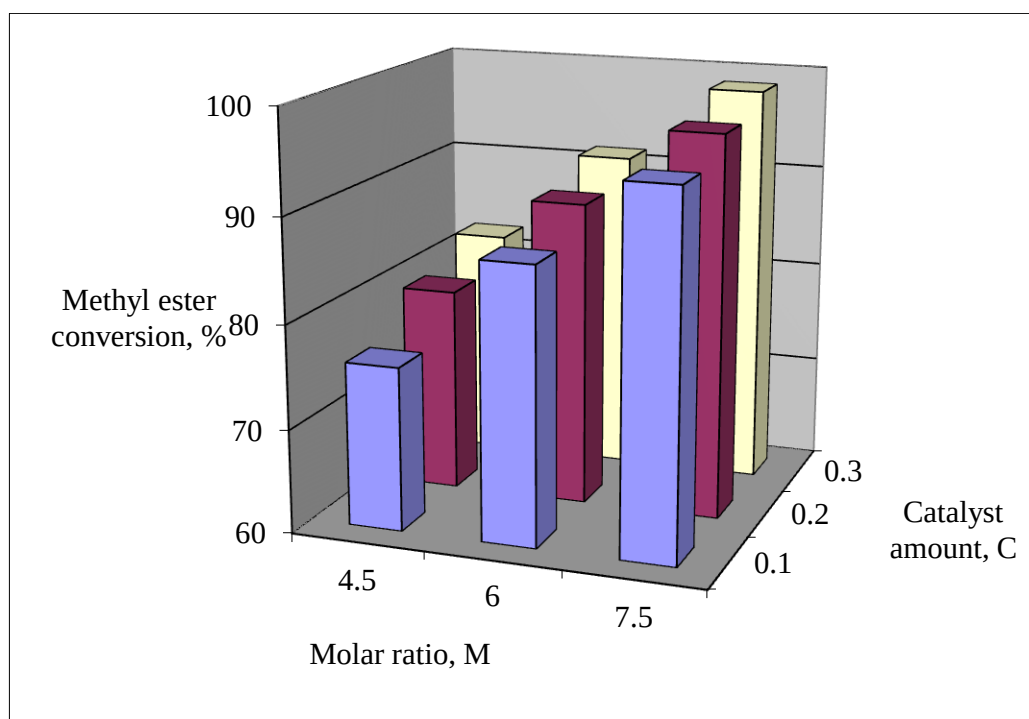


Fig. 4.15. Effect of molar ratio on pungam biodiesel production

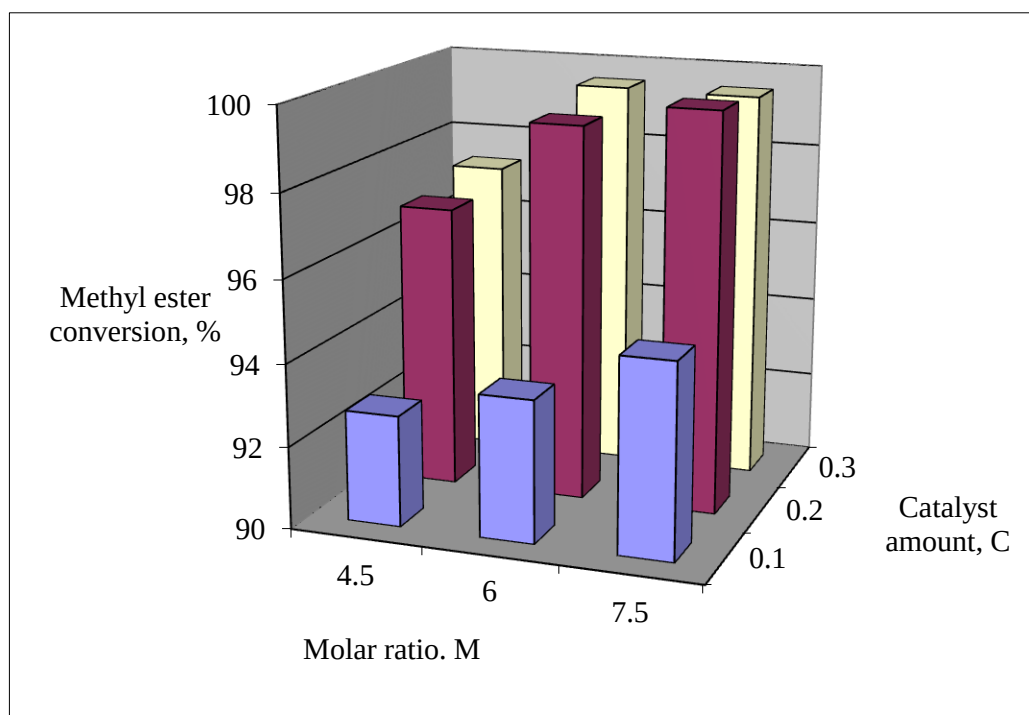


Fig. 4.16. Effect of molar ratio on palm biodiesel production

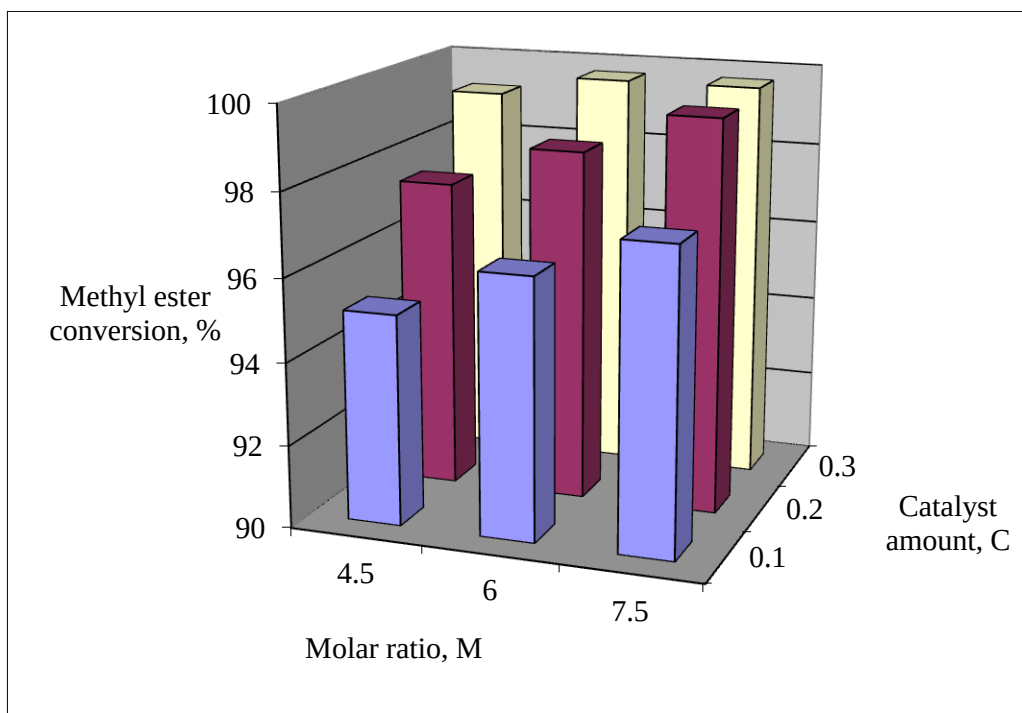


Fig. 4.17. Effect of molar ratio on jatropha biodiesel production

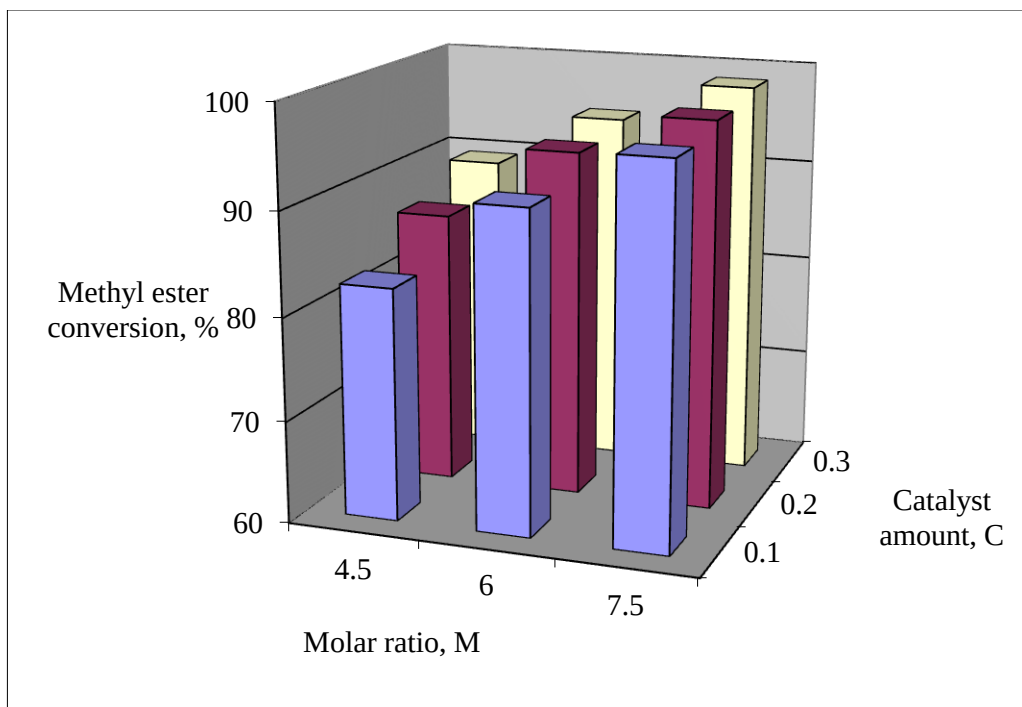


Fig. 4.18. Effect of molar ratio on neem biodiesel production

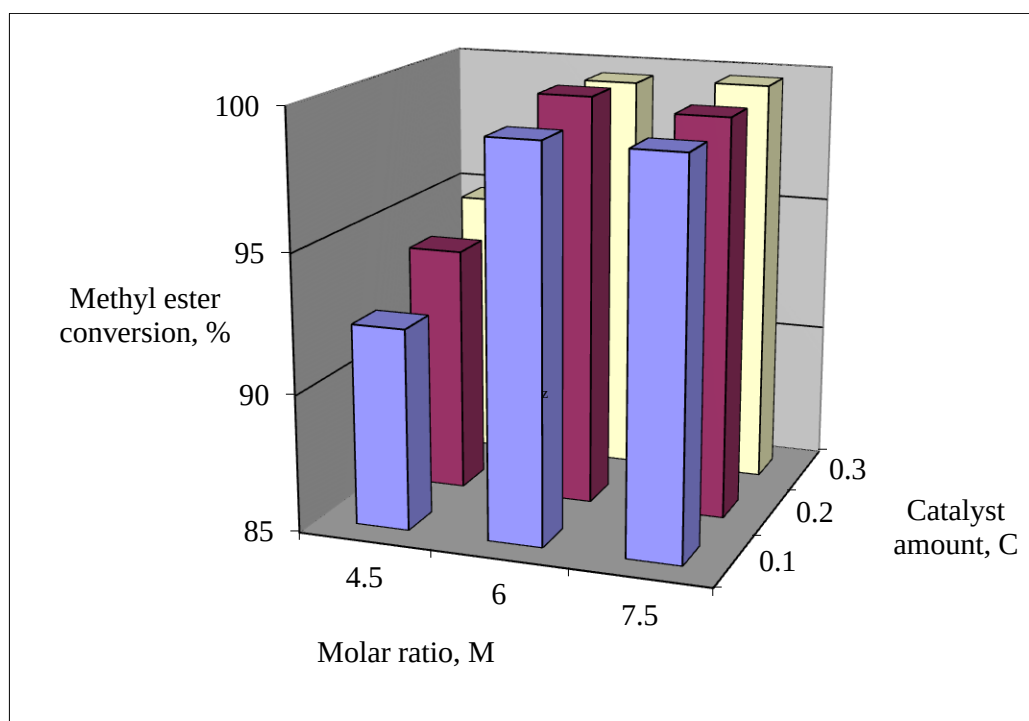


Fig. 4.19. Effect of catalyst amount on coconut biodiesel production

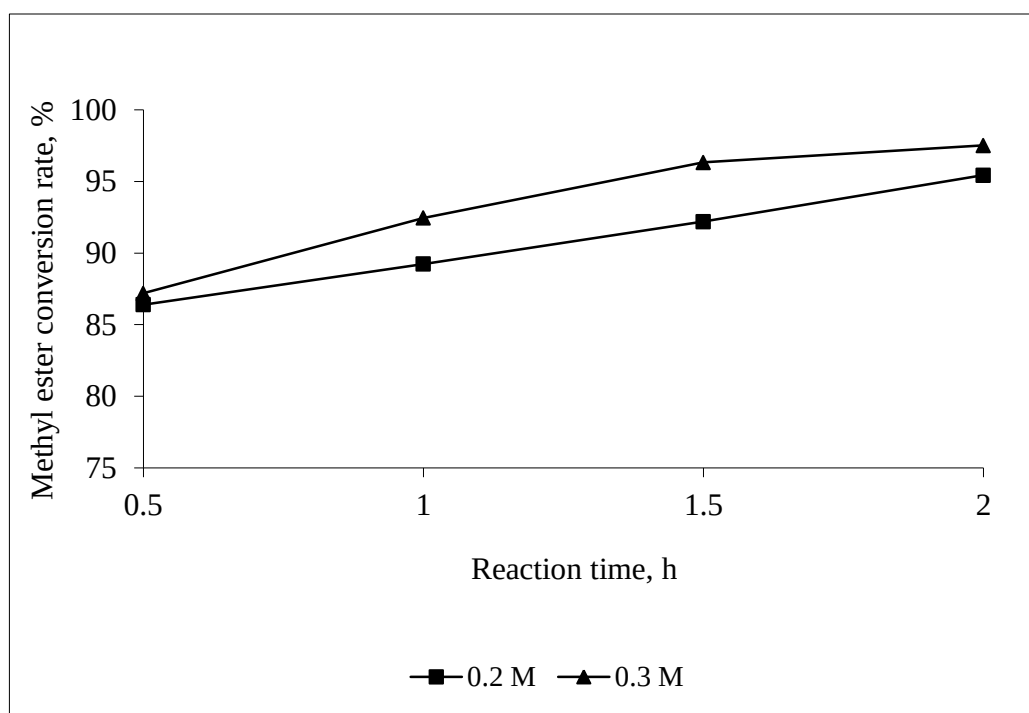


Fig. 4.20 .Methyl ester conversion rate at molar ratio of 1: 5.25 (Pungam oil)

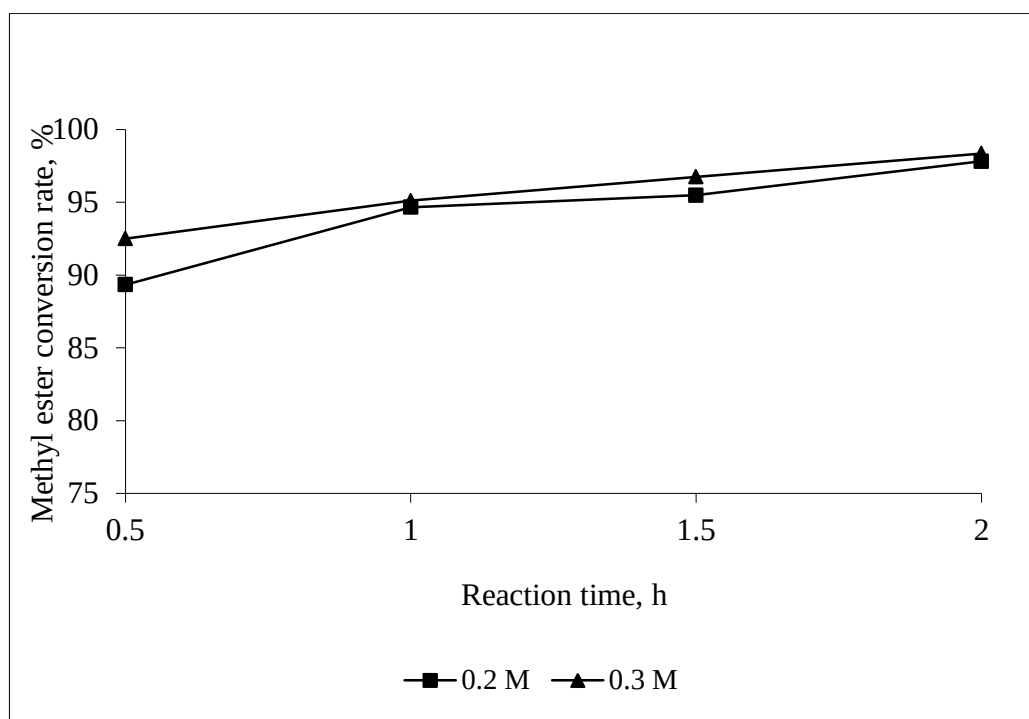


Fig. 4.21. Methyl ester conversion rate at molar ratio of 1: 6 (Pungam oil)

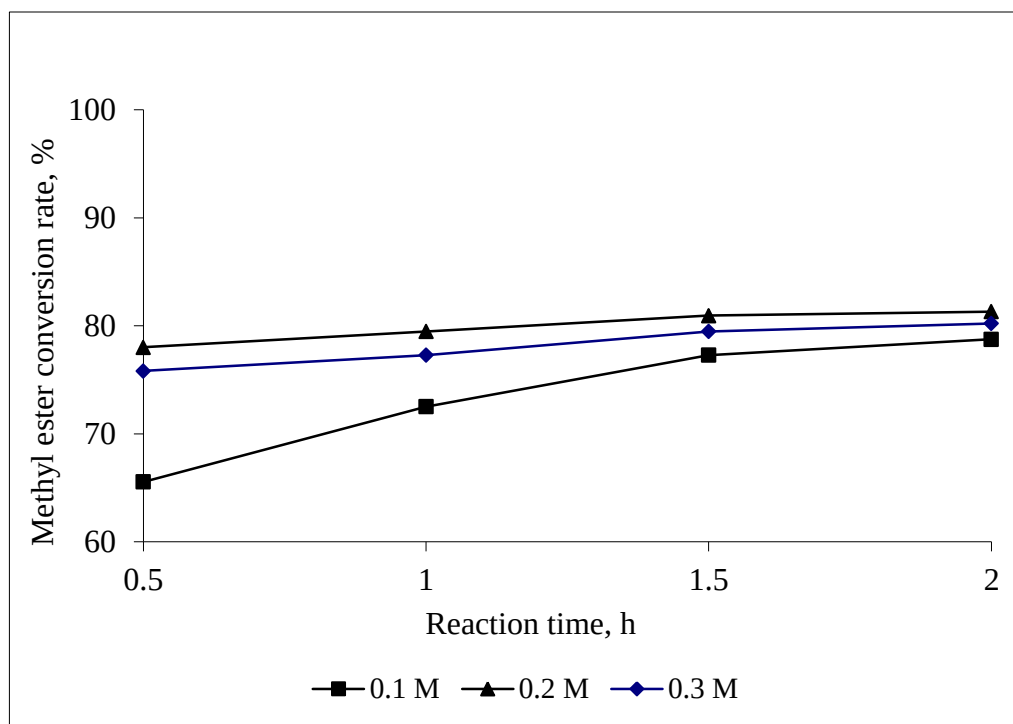


Fig. 4.22. Methyl ester conversion for jatropha oil at molar ratio of 1: 4.5

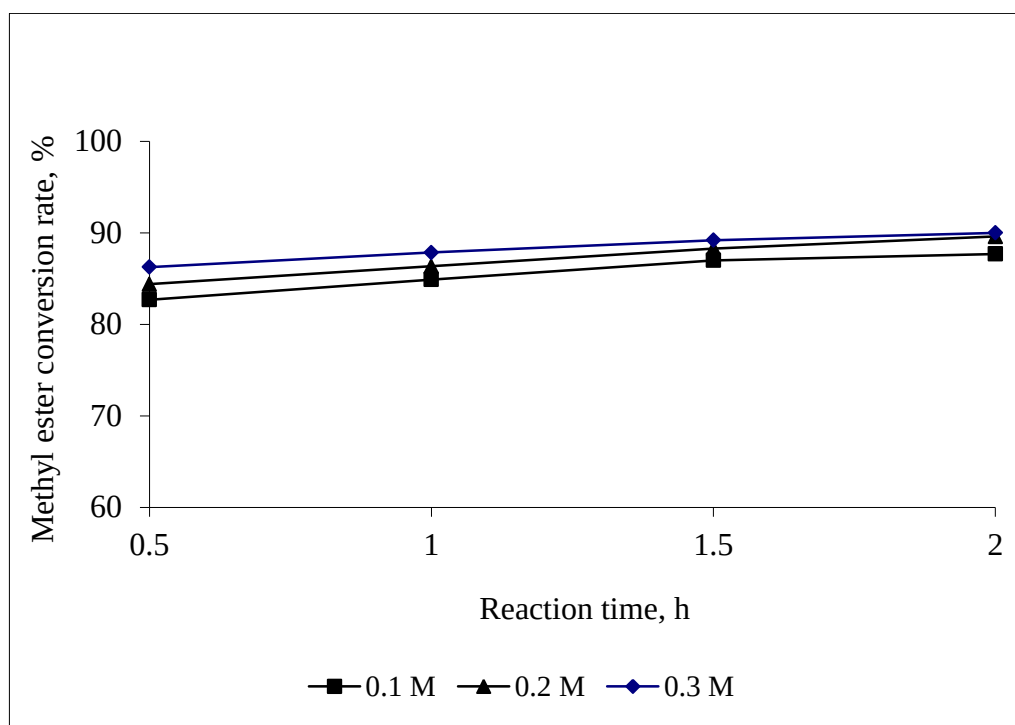


Fig. 4.23. Methyl ester conversion for jatropha oil at molar ratio of 1: 5.25

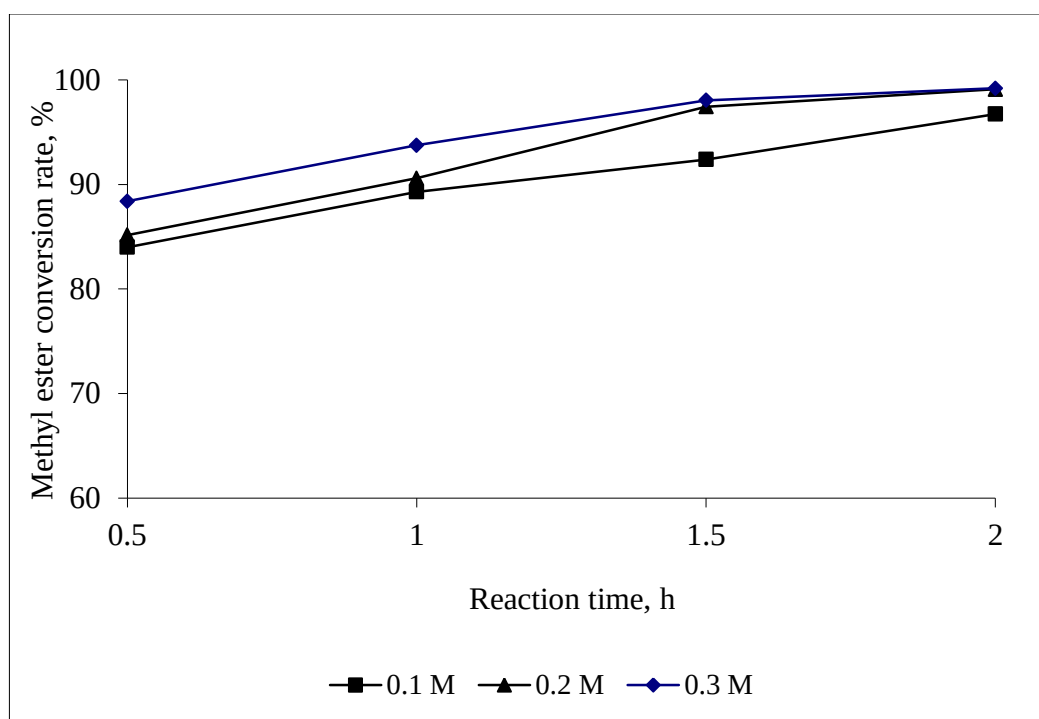


Fig. 4.24. Methyl ester conversion for jatropha oil at molar ratio of 1: 6

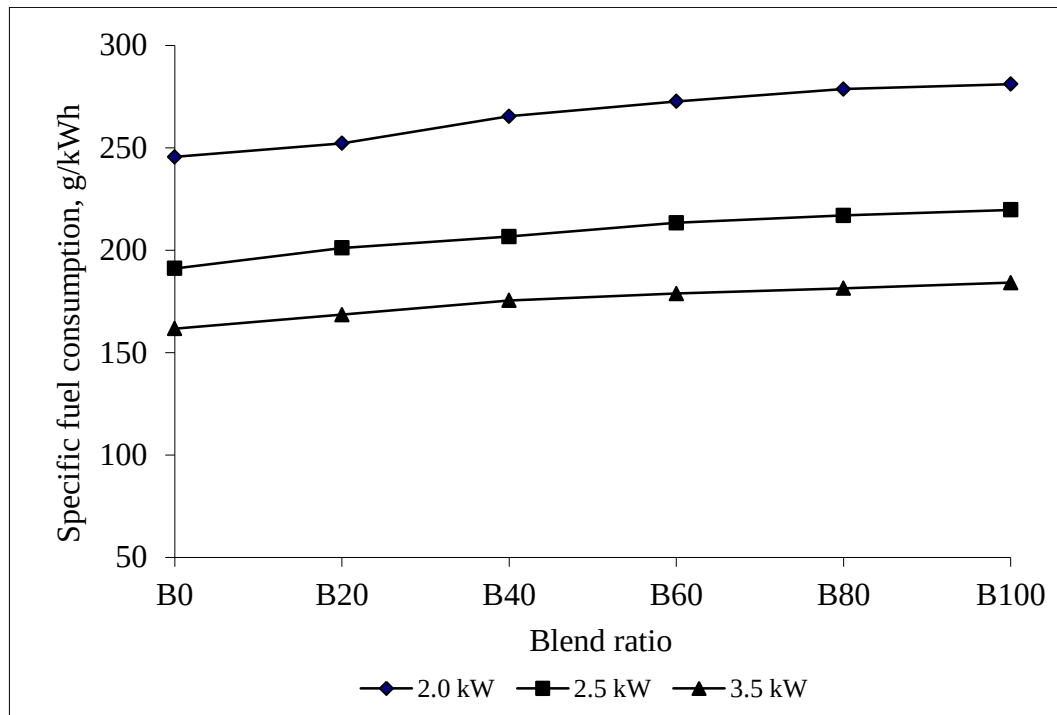


Fig. 4.25. Fuel consumption for biodiesel blended fuels

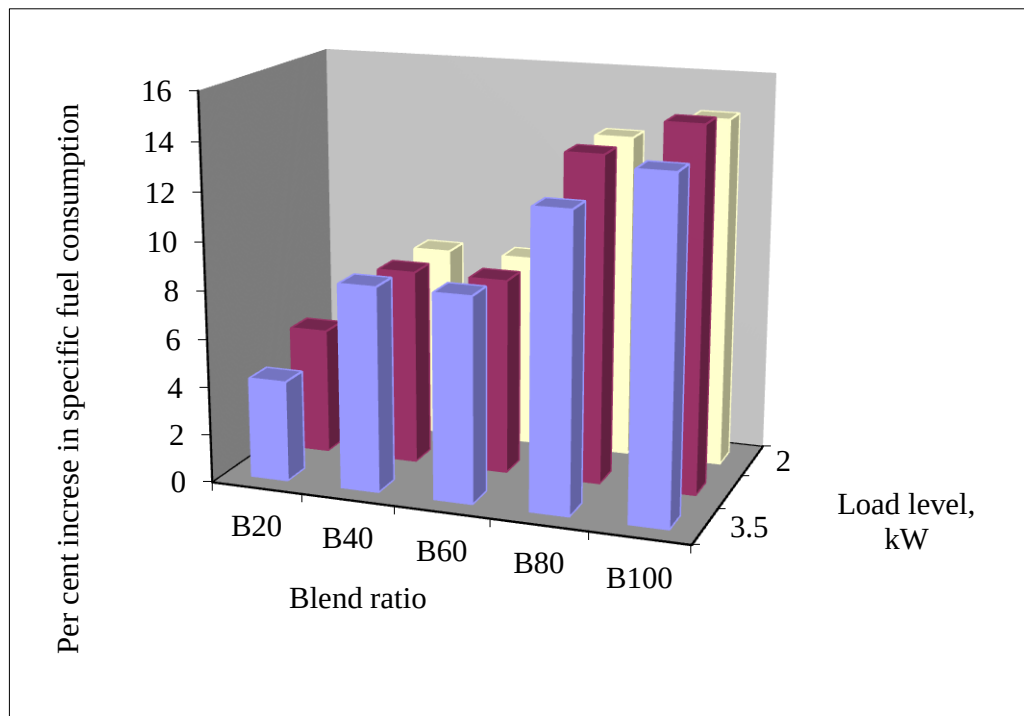


Fig. 4.26. Per cent increase in specific fuel consumption for biodiesel blended fuels

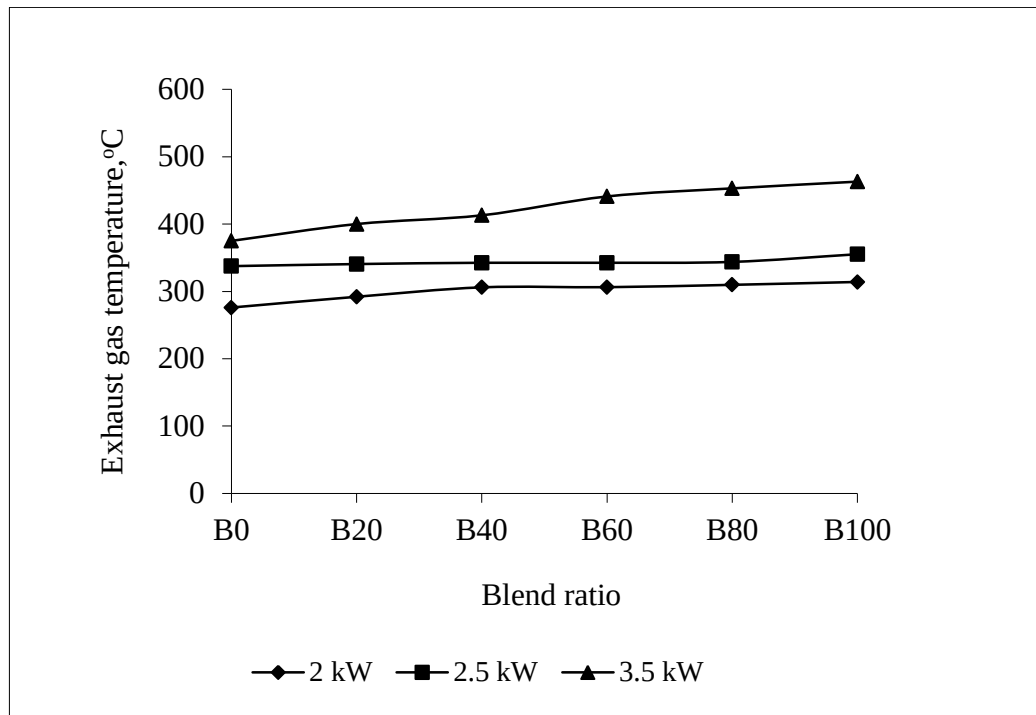


Fig. 4.27. Variation of exhaust gas temperature in diesel engine fuelled with different blended fuels

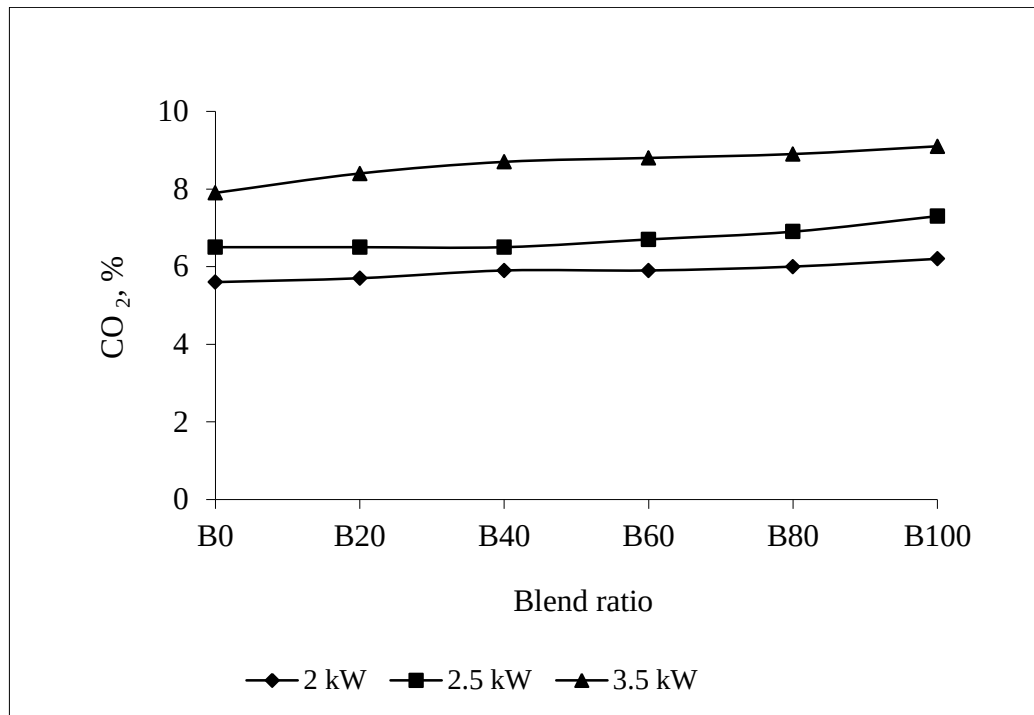


Fig. 4.28. CO₂ emission from diesel engine with different blended fuels

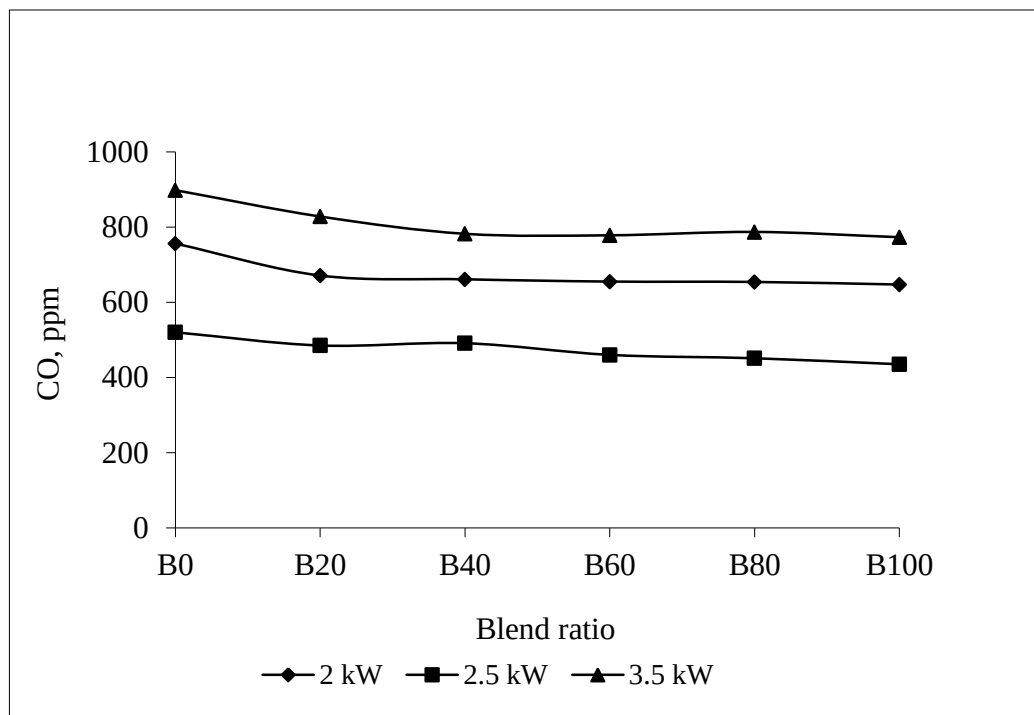


Fig. 4.29. CO emission from diesel engine with different blended fuels

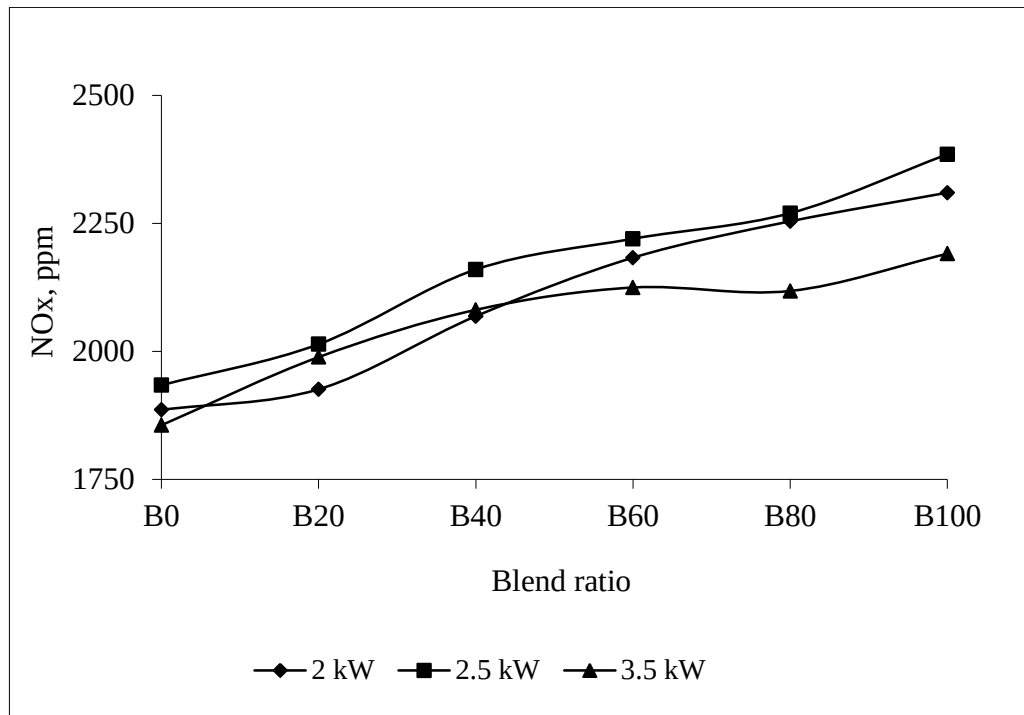


Fig. 4.30. NOx emission from diesel engine with different blended fuels

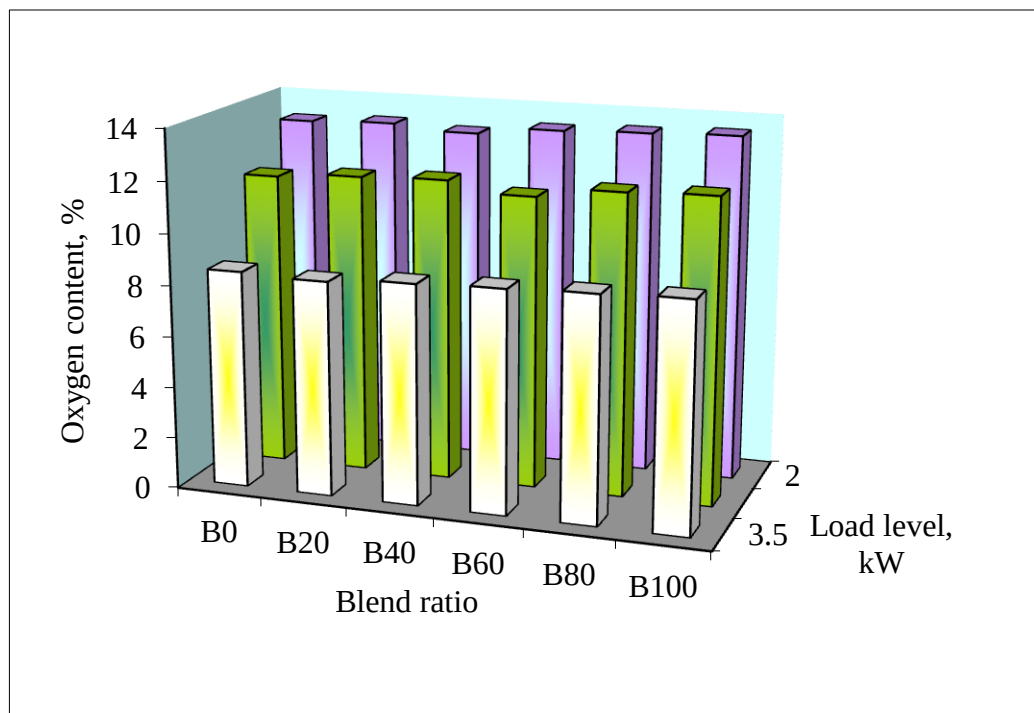


Fig. 4.31. Oxygen emission from diesel engine with jatropha biodiesel blended fuels



Plate 3.1. Pungam tree and seeds

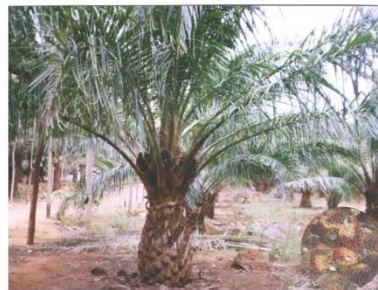


Plate 3.2. Oilpalm tree and fruits



Plate 3.3. Pinney tree and seeds



Plate 3.4. Jatropha tree and seeds



Plate 3.5 Neem tree and seeds



Plate 3.6. Coconut tree and nuts



Plate 3.7. Fuel properties testing instruments



Plate 4.1. Laboratory scale biodiesel reactor



Plate 4.2. Biodiesel washing in separating funnel



Plate 3.8. Gas and High Performance Liquid Chromatograph



Plate 3.9. Elemental analyser



Plate 4.3. Pungam oil and biodiesel



Plate 4.4. Palm oil and biodiesel



Plate 4. 5. Jatropha oil and biodiesel



Plate 4.6. Neem oil and biodiesel



Plate 4.7. Coconut oil and biodiesel



Plate 4.8. Biodiesel pilot plant



Plate 4.9. Jatropha biodiesel and glycerol

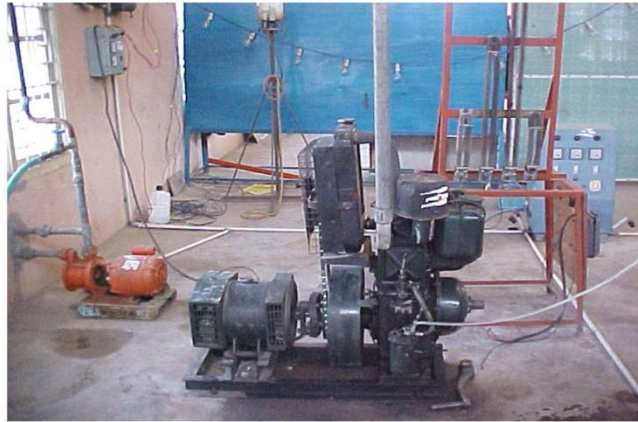


Plate 4.10. Engine testing setup



Plate 4.11. Electrical loading unit