



PLANTATION, PRODUCTION AND USE OF
BIOFUELS
AT THE COMMUNITY LEVEL IN SRI LANKA

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Practical Action

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**Plantation, Production and Use of Biofuels at
the Community Level in Sri Lanka**

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BIODIESEL STANDARDS

ACRONYMS

TAB	Technical Advisory Board
CaME	castor oil methyl ester
CBO	Community Based Organisations
CME	coconut oil methyl ester
CRD	Completely Randomized Design
DMME	Chemistry Department and the Mechanical and Manufacturing Engineering Department
FFA	free fatty acid
IAA	Indol Acetic Acid
IBA	Indol Butric Acid
NERDC	National Engineering Research & Development Centre
RCBD	Randomized Complete Block Design
ROH	University of Ruhuna
SVO	straight vegetable oil
TAG	tri-acyl-glycerol

GLOSSARY

aspirator	an apparatus for drawing out fluids or gases from the body or a body cavity by suction
diglycerides	glyceride containing two fatty acid molecules in ester linkage
esterification	to convert into an ester
ester	formed by reaction between an acid and an alcohol with elimination of water
flash point	The temperature at which a liquid will yield enough flammable vapour to ignite
glyceride	an organic acid ester of glycerol, designated, according to the number of ester linkages, as mono-, di-, or triglyceride
hexane	a colourless liquid hydrocarbon of the alkane series, present in petroleum spirit
hydrophilic	dissolving in, absorbing, or mixing easily with water
hydrophobic	not dissolving in, absorbing, or mixing easily with water
phospholipids	Any of various phosphorus-containing lipids, such as lecithin and cephalin, that are composed mainly of fatty acids, a phosphate group, and a simple organic molecule. Also called phosphatide.
saponification	turning of fat or oil into soap by reaction with an alkali
specific gravity	the ratio of the density of a substance to the density of water
transesterification	a reaction of oil with a primary alcohol in the presence of a catalyst.
viscosity	the property of a fluid or semifluid that causes it to resist flowing
w/w	an abbreviation for "by weight," used to describe the concentration of a substance in a mixture or solution (for instance, 2% w/w means that the mass of the substance is 2% of the total mass of the solution or mixture)

EXECUTIVE SUMMARY

Sri Lanka, like its other South Asian counterparts is a developing country grappling to meet the increasing demand for energy using environmentally sustainable energy sources. Bio-energy provides interesting options to address such issues, with the potential to provide affordable access to transport fuel and electricity for the poor. Practical Action and the Technical Advisory Board (TAB) on liquid biofuels in Sri Lanka endeavour to study this largely untapped and unresearched resource in Sri Lanka under the *Liquid Biofuels* project. The main objective of this project is to demonstrate a community managed biofuel plantation and field test extraction processes. The studies featured in this publication are part of this effort and details research in biofuel plantation, extraction, processing and application.

The initial stage of Practical Action's *Liquid Biofuels* project was to investigate a range of biofuel production potentials and applications as a renewable energy option for the poor in remote off-grid areas of Sri Lanka and elsewhere. To this end the project conducted an audit on the current liquid biofuel status. Practical Action has adopted a self imposed policy not to touch edible crops for producing biofuels, and to promote only non-edible crops for this purpose. As part of the project TAB members have carried out preliminary studies in 4 key phases to find out the suitability of oil bearing seeds grown in Sri Lanka, available oil extraction and processing methods, and to test the application of biofuels and by-products. This publication is a compilation of the findings of this research.

The research institutions involved in this study have expertise in the various research areas assigned to them under this project. The plantation and part of the expelling processes have been undertaken with the assistance of the community. The expelling and processing of the biofuel and the testing of its mechanical application took place in a laboratory environment. Once these techniques are refined they will be introduced to the community with measures taken to ensure high safety and performance - as part of the Stage 2 of the *Liquid Biofuels* project. Apart from the findings of the studies, this publication also contains descriptions of how experiments were conducted. Recommendations based on study results have also been made and a summary of the discussions from a workshop to analyse study findings have also been included at the end of the respective study chapters. Future plans and implementation of the remaining stages of the project are laid out at the end of this book.

1

CHAPTER 1

BACKGROUND

Sri Lanka is a country with a population of approximately 20 million people, most of whom are engaged in farming as their livelihood. Nearly a quarter of the citizens live below the extreme poverty line. Furthermore, the gap between the poor and the rich continues to widen, with the country's Gini coefficient of household income at 0.46 in 2003/2004¹. About 70% of the people live in the rural sector, although there is a shift towards urbanisation. Targeting an economic growth of 10% per year over the next 5 years, Sri Lanka urgently requires alternative forms of renewable energy sources. Escalating transportation and energy costs, and lack of connectivity to the national electricity grid has limited the participation of marginalised communities in the development process. To ensure that this growth spreads to the rural areas and positively impacts the lives of the poor, communities should be provided with adequate access to energy and be equipped to manage the alternative energy options themselves.

Primary Energy in Sri Lanka is supplied predominantly by biomass, hydro and petroleum products. The country is over-dependent on fossil fuels for energy generation and the running of vehicles. The escalating price of such fuels in the global market has hit the poorer societies the hardest. Renewable energy sources in the form of bio-energy show potential. However their potential is yet to be properly investigated and the technology refined. Long term performance studies and initiatives targeting community level implementation for the benefit of the poor are non-existent in Sri Lanka - a gap which Practical Action hopes to help fill, through its Liquid Biofuels project and with the help of its partners and the communities.

1.1 BIOFUEL POTENTIAL AND ENERGY BALANCE

The economic potential for biodiesel processing was estimated in a report prepared by University of Wisconsin, Madison in 2007. Figure 1.1 presents the cost of production (per litre) in different countries divided into 7 bands. The two bands of \$0.29 – \$0.50 and \$0.51 - \$0.70 are recognised as countries with the best potential for economic biodiesel and Sri Lanka falls in the latter band of countries which according to the report is predicted to be able to produce biodiesel at a cost between US\$ 0.51 to 0.70 per litre.

Liquid biofuels come in the form of biodiesel and ethanol through processed plant oil and starch respectively. In Sri Lanka the oil seed potential is yet to be identified. However, some plants which are present in Sri Lanka have been identified in the global market as viable liquid biofuel sources. Biofuels, as a substitute for petroleum products, have been researched in Sri Lanka and particularly in India. However, few explorative researches on biofuels have been conducted in Sri Lanka. Some research institutions have made moves to test the running of vehicles using biofuels while others have attempted growing and testing *jatropha* and a few other crops to extract biofuels.

¹ Annual Report 2007, Central Bank of Sri Lanka.

The potential of future energy generation lies in the use of liquid biofuels for vehicle and cottage industries. Presently the fuel used for vehicles and to produce motive-energy (energy to power vehicles) is diesel or kerosene. The introduction of biofuels to such applications will increase the self-sufficiency of communities in their energy supply. In addition, biofuels are a clean source of energy with a high potential to generate employment for those in remote areas. While the policy for the promotion of bio-energy exists, implementation is slow. No initiatives have been taken by the Government of Sri Lanka to demonstrate the viability of the renewable, community based, liquid biofuel options to attract implementers and end users.

Figure 1.1 Economic potential of countries for biofuels with the best potential for economic biofuels



(Source: University of Wisconsin, Madison 2007)

1.2 THE PROJECT

In order to explore the technical and economic feasibility of using alternative fuel (namely liquid biofuel) for generating power in Sri Lanka, Practical Action formed a Technical Advisory Board (TAB) on Liquid Biofuels in March 2006. The TAB consists of 14 members representing universities, research organisations, financial institutions, entrepreneurs and forest officers, etc. The TAB team made a 5-day biodiesel study visit to Orissa, India to acquire knowledge and know-how on biodiesel production and application at the community level. They visited the projects sites of the Mohuda Pilot Plant and Training Centre to experience how to implement, operate and maintain biodiesel projects in rural communities using the technologies developed by CTxGreEn (Canada) for implementation in core villages of Gram Vikas (one of the largest Indian NGO's with over 28 years experience in Orissa).

Using the experience and know-how gained from the study visit as well as their own expertise TAB carried out preliminary studies in 4 key areas to find out the suitability of oil bearing seeds grown in Sri Lanka, available oil extraction methods, and the processing and application of biofuels and by-products. TAB, Practical Action and community partners used the information generated from the above initiatives to set up a community level demonstration on biofuels plantation, expelling and applications – know as the Liquid Biofuels project. Having

commenced in April 2006, the project's pilot test is being conducted in the rural community of Gurugoda in Rasnayakapura Divisional Secretariat, Sri Lanka. The overall goal of the project is to research, demonstrate and influence the relevant stakeholders on eco-friendly solutions to help resolve energy and transport difficulties faced by the poor in a sustainable, community based manner. This publication is a compilation of the findings of research undertaken in the aforementioned project with regard to plantation growth, extraction, processing and application of biofuel via rural communities.

The main objective of the Liquid Biofuels project and its stakeholders is to demonstrate a community managed biofuel plantation and field tested extraction processes.

The project's main goal is to conduct performance studies of community managed bio-energy systems and demonstrate the potential for fuel plantations in Sri Lanka. Enhanced performance, techno-social improvements to reduce costs and increase income generating options, as well as assessing the future potential of these technologies is the main aim of this project. Approximately 30 families are involved in the project and will benefit from the income generated from proposed initiatives.

Community involvement within the project is highlighted as it is closely linked to the social and economic sustainability of proposed initiatives. Targeted for this ground breaking demonstration is the community of Gurugoda in Rasnayakapura Divisional Secretariat and the Nikaweratiya Pradeshiya Sabha. This community is the key stakeholder of the project. It is a village with which Practical Action has previously worked and within which it has succeeded in building an effective community governance structure. Community roles consist of that of growers and managers of the plantation and processing activities. The community has formed a specific Sub-Community Based Organisation (CBO) responsible for managing the plantation and fuel extraction processes as well as taking part in energy and transport applications.

Governmental and non-governmental institutions are involved in the project, conducting research and supporting the village in the planning, management and maintenance of this project. Nikaweratiya Pradeshiya Sabha and Rasnayakapura Divisional Secretariat (local authorities) play mainly an administrative role, and assist in linking the project to other government agencies which may be interested in project results. TAB acts mainly as an overall advisor to the project. It provides guidance and technical inputs while Practical Action ensures that the project is in line with national interests. In addition, this national body provides links to the main stakeholders and interested audiences in the biofuel sector to share project results and exchange experiences. The universities of Peradeniya, Moratuwa, Ruhuna and other research institutions are involved in the research aspects in engineering and agro-forestry which are linked to the project outcomes in the form of monitoring and providing research inputs. Sangrama co-ordinates implementation at community level.

PROJECT STAGES

Stage 1

Initial testing and research – This stage involved the study of seed cultivation, extraction, processing and biofuel application. Experts assisted communities to plant specific plants which were already readily available in the area, but were not utilised or formerly cropped. Subsequently the seeds were harvested and then taken to the laboratory for testing. The seeds are then processed and tested to generate biofuel and the fuel was finally tested on appropriately adjusted vehicles/machinery. To ensure the studies were comprehensive, other types of seeds were also tested to ascertain their possible potential for biofuel generation.

The research and information generated from the above processes is what is represented in chapters 2 - 5 of this publication.

Stage 2

Establishing an energy plantation (mixed plantation) at household level – The information gathered from the research conducted in stage 1 was used to help communities establish mixed plantations of biofuel crops grown at household level. These crops were grown and harvested by the families. Approximately 30 families were targeted for the demonstration in order to ascertain the systems which work in such crop plantations.

Extraction and Processing of bio-diesel - The processing of biofuel was undertaken by the partner organisations and the Liquid Biofuel Centre technicians with the assistance of technical officers from Practical Action and other stakeholders. The machinery is being maintained and managed by the CBO in the village. Sharing of returns from extracted biofuel between planters was monitored and documented as were the extraction, inputs and output quantities.

Stage 3

Biodiesel Applications (testing of transport, water pumping, electricity generation with biodiesel) – At the time of going into publication the testing of the processed biofuel was to be undertaken mainly by the research staff and technical officers, with Practical Action providing technical support. The testing involves assessing energy generation for vehicular use, water pumping, and generator mechanisms. Existing small scale engines will be used in the testing process and the conventional fuels will be (partially or fully) replaced with biofuel and the necessary technical adaptations made. The performance of these different engines with this fuel replacement and other necessary information on such adaptation, such as the costs incurred, will be documented.

Throughout the project, documentation of the project processes, experiences, testing and results is undertaken by stakeholders as well as Practical Action. At the end of the project a publication containing the outcomes of this community based initiative in biofuel (a first in Sri Lanka) will be published. This will be

a follow-up publication to the 'Plantation, Production and Use of Biofuels at the Community Level in Sri Lanka'. The socio-economic development of the community, assessment report on resulting yields, seasonality of yields and biofuel extraction and application processes within the community will be included in the second publication.

1.3 THIS BOOK

'Plantation, Production and Use of Biofuels at the Community Level in Sri Lanka' is the result of the research, testing and subsequent information generated from Stage 1 of the Liquid Biofuels project. Practical Action provides the overall leadership and co-ordination of the project activities while TAB members provide guidance and technical inputs. It is a compilation of the results and processes undertaken in the 04 key sections of harnessing biofuel; plantation, oil expelling, processing and application. The research institutions involved in this study are members of TAB and, as such, have expertise in the various research areas assigned to them in this project. The plantation and part of the expelling processes have been undertaken with the assistance of the community. The expelling and processing of the biofuel and its mechanical application took place in the laboratory. Once these techniques are refined they will be introduced to the community with measures taken to ensure high safety and performance as part of the Stage 2 of the Liquid Biofuels project.

This publication is a compilation of 4 reports submitted by those TAB members who undertook the studies. Research into an energy plantation for biofuel was headed by Professor S. Subasinghe at the University of Ruhuna. Planting jatropha seeds - two varieties for the dry and wet zones respectively – his team analysed the crop yield and susceptibility to disease. With regard to oil expelling Ms. Y.M. Malini. K Ranatunga from National Engineering Research & Development Centre (NERDC) details the basic processes involved in oil expelling which consists mainly of seed cleaning, seed drying, extraction, filtering, packaging and storage of oil. In her experiments the seeds used for extraction were castor, jatropha, domba, rubber and neem. Dr. C. S. Kalpage from the University of Peradeniya researched biodiesel processing from vegetable oils. Besides those received from NERDC from the oil expelling stage of this study other vegetable oils were also tested by his team, the results of which are contained in this publication. Dr. H.C. Ambawatte from the University of Ruhuna tested the transesterified jatropha oil samples and its different blends with diesel tested on engines. The density, specific gravity, viscosity and flash point of different blends were determined and finally their viability for application as fuel in engines was assessed.

The findings of the above studies were discussed by TAB members at a two day workshop (held on the 23rd and 24th of March 2008). Outcomes of this workshop and the discussions pertaining to each study are represented at the end of the chapters dealing with each particular study. Future plans and implementation of the remaining stages of the project are also laid out at the end of this book.

2

CHAPTER 2

ENERGY PLANTATION FOR BIOFUEL

By Professor S. Subasinghe, University of Ruhuna

2.1 INTRODUCTION

A community based energy plantation (consisting of non-edible liquid biofuel crops) was established in Rasnayakapura Divisional Secretariat as a pilot investigatory phase in the biofuel plantation. To this end, Professor S. Subasinghe and his team at the Faculty of Agriculture, University of Ruhuna studied the propagation and field performance of jatropha as part of the first study under the Liquid Biofuels project. Seed germination studies of two jatropha varieties (*Jatropha curcus* and *Jatropha abosa*) were conducted at the University of Ruhuna while individual nurseries of *Jatropha curcus* were also set up in farmers' home gardens at Rasnayakapura.

At the University of Ruhuna: A research plot of both varieties of jatropha was established at the University premises. Studies on the propagation of *Jatropha curcus* was completed at the Faculty of Agriculture, University of Ruhuna under the final year student research project during March-August 2007.

At Gurugoda, Rasnayakapura: 13 families were trained on the application of seed treatments, preparation of cuttings, and planting and maintenance of a nursery. The families used the seeds which were collected from their surroundings. Households were paid Sri Lankan Rs. (LKR) 18/- per plant for planting and maintenance (out of which LKR 5/- went to the community organisation).

Introduction to Jatropha

Botanical Description:

Family - Euphorbiaceae, Genus - *Jatropha*, Species - *Jatropha curcus*

Jatropha is a perennial plant having a lifespan of 50 years or more. A tree starts bearing seeds typically within 2 years, but the maximum yield occurs after 4-5 years. *Jatropha* plants can be grown on a wide range of soil and climatic conditions. They can grow very well in wastelands, along the canals, roads, railway tracks, on the borders of farmers' field as living fences, etc.

Jatropha is mainly used for extracting oil from the seeds. About 3-4 kg of *jatropha* seeds are needed to produce 1L of *jatropha* oil. The oil is also used for making soap, lightening lamps, etc. The bark of the *jatropha* plant contains "Jatropings", which have anti-cancerous properties. Roots

are used as an antidote for snake bites. Leaves are used for fumigating houses against bed bugs. Glycerine is obtained as a by-product from the plant.

2.2 THE STUDY

No research studies have been conducted locally on the propagation and cultivation methods of jatropha. *Jatropha* is mainly propagated by seeds and stem cuttings. There are several limitations in seed propagation. A hard seed coat and low viability are the main problems. Thus, people are not willing to cultivate *jatropha* as a commercial cultivation. Therefore, a series of experiments were conducted in this study to induce germination of seeds and propagation of *jatropha* through stem cuttings at the Faculty of Agriculture, University of Ruhuna.

Four experiments were conducted in this study. They mainly focused on the seed treatments and storage conditions and their effect on seed germination, in vivo propagation studies done through stem cuttings, and the study of growth performance as affected by different spacing.

EXPERIMENTS

EXPERIMENT 1:

SEED GERMINATION OF *JATROPHA CURCUS* AS AFFECTED BY DIFFERENT SEED TREATMENTS

The following seed treatments were used.

Hot water treatment:	50 °C for 1 min.
	50 °C for 3 mins
	60 °C for 1 min
	60 °C for 3 mins
	70 °C for 1 min
	70 °C for 3 mins
Seed soaking:	12 hours
	24 hours
Seed clipping	
Seed scarification	
Control (Normal seeds)	

The experiment was arranged with Factorial Completely Randomized Design (CRD) with 3 replicates, and the germination percentage was recorded daily.

RESULTS FROM THE DIFFERENT SEED GERMINATION TREATMENTS:

- Seed clipping was the best seed treatment
- Hot water treatment had no effect on seed germination
- No significant difference in water soaking treatments on seed germination

EXPERIMENT 2:

GERMINATION OF JATROPHA SEEDS AS AFFECTED BY STORAGE CONDITIONS

Storage conditions: Room temperature
Cool temperature (4 °C)

Experimental design was Factorial CRD with 4 replicates.
Seed germination percentage was recorded daily

RESULTS:

Seed germination decreased with increasing periods of storage. Storage at cool temperature extends the seed's viability (up to 4 months of storage) than storing seeds at room temperature.



Photo 1: Seed germination after 4 months of storage at room temperature



Photo 2: Seed germination after 4 months of storage in refrigerator

EXPERIMENT 3:

IN VIVO PROPOGATION OF JATROPHA (*Jatropha curcus*) THROUGH STEM CUTTINGS

Treatments:

Cutting types:	Soft wood cuttings Semi-hard wood cuttings Hard wood cuttings
Rooting media:	Sand 1:Compost 0 :Top soil 0 Sand 1:Compost 0 :Top soil 1 Sand 1:Compost 1 :Top soil 1
Rooting Hormone:	Indol Acetic Acid (IAA) Indol Butric Acid (IBA)

RESULTS

Semi-hard wood cuttings in media containing sand, top soil and compost (1:1:1) with IBA hormone showed the highest rooting followed by soft wood cutting in the same medium which was not significantly different.





Photo 3
Hard wood cutting with IAA



Photo 4
Semi - hard wood cuttings with IBA

SHOOT VIGOUR OF PLANTS



Photo 5

T1

T2

T3

T4

T5

T1 – Soft wood cuttings with IAA in sand media

T2 – Hard wood cuttings with IAA in sand media

T3 – Semi-hard wood cuttings treated with IAA in sand media

T4 – Soft wood cuttings treated with IBA in sand, top soil and compost – 1:1:1 media

T5 – Semi-hard wood cuttings treated with IBA in sand, top soil and compost – 1:1:1 media

Soft wood/semi-hard wood cuttings in sand, top soil and compost – 1:1:1 media - with the hormone IBA, showed significantly higher shoot vigour (T4).

EFFECT OF ROOTING MEDIA AND HORMONE



Photo 6 *IAA with Sand*

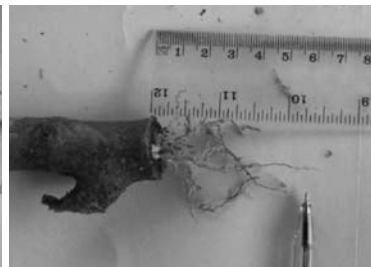


Photo 7 *IBA with Sand*

RESULTS:

Soft wood/ Semi-hard wood cuttings treated with IBA rooting hormone and planted in sand: top soil: compost – 1:1:1 media - showed significantly higher rooting (root fresh weight and higher number of roots).

EXPERIMENT 4:

EFFECT OF PLANT SPACING ON GROWTH PERFORMANCE OF FIELD ESTABLISHED JATROPHA

Treatments used (Spacing)

90cm x 60cm

90cm x 90cm

90cm x 120cm

Following data was recorded

Number of shoots

Number of leaves

Shoot height

Time taken for flowering

EXPERIMENTAL DESIGN:

The experiment was conducted in a Randomized Complete Block Design (RCBD) with 4 replicates.

Experiments are still going on and growth and yield data were collected for a one year period after planting. In *Jatropha curcus*, the average number of leaves and average plant height was 18 cm and 21 cm respectively after 5 months, and 42.3 cm and 59.2 cm respectively in one year. The average number of leaves and plant height of *Jatropha abosa* variety were 103 cm and 47 cm in 5 months and 111.5 cm and 76.7 cm in one year respectively (Table 2.1).

Table 2.1:
Growth parameters at 5 months and one year after planting in field at University of Ruhuna

Months after planting	Leaf number per plant		Plant height / (cm)	
	<i>Jatropha curcus</i>	<i>Jatropha abosa</i>	<i>Jatropha curcus</i>	<i>Jatropha abosa</i>
5	18.2	103.0	21.0	46.6
12	42.3	111.5	59.2	76.7

Mealy bug is a common pest incidence for most of the crops. The insect sucks the juice of the plant and ultimately retards plant growth, and finally the entire plant can die. This pest can transmit virus diseases as well.



Photo 8 Mealy bug damage for Jatropha curcus Plantation in Ruhuna (5 months after planting)

2.3 PROCESS OF PLANTING JATROPHA IN NIKAWERATIYA



Photo 9: Preparation of Polybags (Sealing)



Photo 10: Filling of polythene bags for planting



Photo 11: Cuttings and seeds are planted in polythene bags



Photo 12: Polybag nursery of Jatropha



Photo 13: Well grown *Jatropha* seedling ready for planting



Photo 14: *Jatropha* Seedlings, planted along the fences

During the 2nd week of October 2007 13 farmers established *jatropha* plants (which were raised in the polybag nursery using seedlings as well as cuttings) along their fences. The following growth parameters were observed from the fences at Gurugoda.

**Table 2.2:
Growth parameters at 5 and 8 months after planting in Gurugoda**

Months after planting	Average leaf number per plant		Average Plant height / (cm)	
	Seedlings	Cuttings	Seedlings	Cuttings
5	58.72	46.08	23.5	22.45
8	81.67	61.20	61.2	49.4

Growth rate of *jatropha* in Gurugoda was much higher than at the *jatropha* plantation in Ruhuna University. 5 months old seedlings had an average plant height of 23.5 cm and 58.72 leaves. Plants propagated through cuttings in the same growth period showed a little less height (22.45 cm) and less number of leaves (46.08 leaves) since the growth of cuttings was poor.

2.4 CONCLUSION

2.4.1 Conclusions from the study

Propagation studies

1. Seed clipping is the best seed treatment in order to achieve the highest seed germination.
2. Seed viability decreases with increased storage periods of seeds.
3. Storage at cool temperature can extend the seed viability up to 4 months than storage at room temperature.
4. The best potting media is either sand: top soil -1:1- or sand: top soil: compost -1:1:1 - for both seed and vegetative propagation.
5. The best cutting types are both semi-hard wood cuttings and soft wood cuttings
6. Best rooting hormone is IBA.

Field studies

1. So far seedlings are the best planting material for field planting in both the wet zone as well as the dry zone.
2. *Jatropha curcus* seedlings grew well in the dry zone (Gurugoda)
3. *Jatropha abosa* seems to be a promising variety as far as growth parameters are concerned for the wet zone when compared to *Jatropha curcus*, which had poor growth in the university plot at the initial stages. However, when considering the growth and yield performance of a 14 months old field plantation, this variety also seems not to be promising in the wet zone of Sri Lanka.
4. Some pest problems occurred affecting plants at the field in the University of Ruhuna (severe attack of mealy bug).
5. 50% of the *Jatropha curcus* plants at the University of Ruhuna died due to waterlogging which occurred as a result of several months of continuous heavy rains. Therefore it is clear that *Jatropha curcus* seedlings do not seem to be so tolerant to waterlogging, especially during the early growth stage.
6. *Jatropha abosa* started fruiting early (5 months after planting) and has no season for fruiting. But seed yield is very low when compared with *Jatropha curcus*
7. Some incidence of disease was found in the Gurugoda plantation. It is suspected that it was a virus disease which also affected the nearby Cassava plants.
8. Except for a few pest and disease incidents, jatropha can be grown well in Gurugoda (dry zone) if managed well, particularly during the early stage of growth. However, continuous observations need to be made for at least another 2 or 3 years to draw concrete conclusions.
9. When considering income and other socio-economic factors, large scale jatropha cultivation may only be possible as either mixed cropping or growing as live fences rather than monocropping.



Photo 15: Pest attack (Leaf rolling caterpillar damage) of *Jatropha* Plants at Gurugoda (3 months after planting)

2.4.2 Outcomes of study assessment at the Biofuel Workshop

Professor S. Subasinghe made a presentation at the Biofuel Workshop on the research he undertook on the energy plantation, where 2 varieties of jatropha were used, a seedling nursery was maintained at the University of Ruhuna and seedlings and cuttings planted in Gurugoda. Seed propagations was done using different seed treatments (e.g. seed clipping, soaking, hot water treatment, seed storage period, cutting type, potting media and two types of hormones (IAA and IBA). For the experiments in Gurugoda, 13 farmers participated in the planting of the seedlings – 400 seedlings and 400 cuttings were planted along their fences. Seedlings have grown well compared to vegetatively propagated plants and most of the plants are now at the bearing stage.

A virus infection was seen in some jatropha plants in Gurugoda which can be seen on Cassava plants too. No pest incidents were observed at the Gurugoda plantation.

The main outcomes of this study were as follows:

- The problem faced in the young plantation at the initial stage was the lack of adherence to the instructions given to the farmers. The seedlings and cuttings were initially planted along the fence and maintenance varied between the farmers.
- Seed bearing started in some plants 5 months after planting in Gurugoda (not in 2 years as found in the literature).
- Seeds could be stored for approximately 4 months without significant loss of viability/germination.
- *Jatropha abosa* grows better than *Jatropha curcus* in the Wet Zone at the initial stage but after about one year growth performances did not seem to be promising
- It was observed that seed propagation was better than propagation through cuttings.
- It was also noted that the village children who participated in the study were asked to maintain records on plant growth etc, and they showed great enthusiasm during the entire study. This may be a good option for monitoring and implementation in future initiatives.
- There is a lot of bare land in the village and farmers cultivate crops only for *Maha* season (rainy season) and abandon the land for the rest of the period. So farmers can establish jatropha as a mixed cropping with some other crops, farmers can use their lands more productively and they can earn income during off-season from jatropha.

DISCUSSION

The following points were voiced during the workshop discussion on the energy plantation study.

- It may be correct to assume that jatropha is more competitive to other crops (in other words absorbing nutrients faster) and may therefore compete with other crops better for nutrients. Thus, in the case of mixed cropping, adequate spacing may need to be provided to give all plants access to adequate nutrients.
- In mixed cropping there may also be a decreased risk of the passing of diseases.
- It was highlighted that the purple variety of jatropha (*Jatropha abosa*) was not seasonal. In other words, it grows throughout the year and therefore can be extracted/harvested continuously. This could result in a better cash flow and encourage farmers to start commercially planting the crop.
- It was noted that inter-cropping is a must if farmers are to receive a continuous cash flow, and it was suggested that when mixed cropping took place it was better to use crops which are familiar to farmers. This will reduce the interference/disturbance to their existing cropping pattern and facilitate the introduction of the cropping of jatropha into their farming system.
- The market system has to be in place to encourage farmers and make them look after their crops (example: a fixed market price).
- Weeds may pose a problem and growth is retarded drastically if weed control measures are not adopted at the early stage of growth in a jatropha plantation.

POSSIBLE FUTURE STUDIES IDENTIFIED

- The cost factors and yield of the two varieties of jatropha need to be compared.
- It was also suggested that other varieties or clones (cross-breeding) crops which may have increased oil production etc. also need to be identified in future.
- An economic assessment of the product needs to be undertaken. The percentage of oil which can be extracted and its value compared with the profitability of growing other crops such as tea/rubber needs to be assessed. The cost of inputs vs. outputs as well as the land suitability should also be assessed.
- As jatropha grows naturally in certain areas of the country, it may be useful to get an idea of the yield of existing jatropha plants (not only that of a pilot plantation).
- Lighting/shade, soil conditions and other environmental conditions which would increase yield should be investigated.
- Pollination – mainly self-pollination – needs to be studied further.
- The fertiliser value of the oil cake produced as well as other income generating aspects and benefits to the community and households should be studied and highlighted to planters.
- Investigate the use of water, and fertiliser in biofuel plantations.

3

CHAPTER 3 OIL EXPELLING

By Ms. Y M Malini K Ranatunge, NERDC

3.1 INTRODUCTION

The second study involves the expelling of oil from the seeds provided from jatropha and other plant varieties. The oil was extracted and tested for processing into biofuel.

The non-edible seeds used for oil extraction were Castor (*Ricinus communis*) Jatropha, (*Jatropha curcas*), Rubber (*Hevea brasiliensis*) and Neem (*Azadirachta indica*). All seeds were supplied by Practical Action and raw material preparation, expelling and oil separation were done by the NERDC at their laboratory in the Agriculture and Post Harvest Technology Department, under the guidance of Ms. Y.M. Malini K. Ranatunge. The basic processes involved in oil processing consisted of seed cleaning, seed drying, extraction, filtering, packaging and storage of oil.

Not all the supplied seeds were used for oil extraction as about 25% of the rubber seed and 2-3% of other seeds received were in a deteriorated condition. After extraction two end products - i.e. crude oil and press residues - were quantified and the yield of oil and remaining oil in press residues were determined. The practicing methods and techniques of oil extraction for different kinds of seeds were identified and areas for improvements were noted.

Evaluating the possibilities of seed oil extraction using a commercially available oil expeller was also a part of the project. Although most of the commercially available oil expellers use three phase electricity, only a single phase power supply is available at the Gurugoda biodiesel site. Thus, a single phase unit with highest capacity in the market was selected, i.e. expeller with a 5 HP motor.

In addition to the processing of seeds given by Practical Action, some other types of seeds such as domba (*Calophyllum inophyllum*) and karanda (*Pongamia pinnata*) seeds were also processed. Some of these resulted in very good oil yields. Castor, jatropha, rubber, neem, domba and karanda oils were extracted mechanically as this method was found to be more convenient than using the chemical method. The highest oil yield of 60% (w/w) was obtained with domba seed while the lowest 8% (w/w) with karanda.

The pressed cake (poonack) produced during the oil expelling process was also tested to quantify the remaining oil in it. Results from testing

biogas production from neem pressed cake and rubber pressed cake at a laboratory scale are described below. Also described are the basic processes involved in oil processing: seed cleaning, oil extraction, filtering and packaging and storage.

Processed seed oil was handed over to the Department of Chemical and Process Engineering, University of Peradeniya for conversion into biodiesel (conversion study discussed in Chapter 4).

3.2 SEED COLLECTIONS AND SUPPLY

3.2.1 Collection Procedures followed by Practical Action.

Seed collecting centers are available in almost every district in Sri Lanka for Ayurvedic medicinal purposes. Seeds were purchased from such collection centers for the study's initial expelling purposes (except rubber seed). At the initial stage, there were difficulties in purchasing good quality seeds due to the lack of proper collecting channels and producers. Seeds which had been collected had some impurities such as sand, small stones, woodchips and spoiled seeds. Prior to extraction, all the foreign particles were removed from the collected seeds.

3.2.2 Seeds

At the initial stage of the project, the selected non-edible seeds and quantities were as follows:

Variety of seed	Supplied quantity (kg)	Expelled and filtered oil quantity (litres)	Remarks
Castor	200	52	
Jatropha	47.5	6.0	
Rubber	350	36.5	500 kg just harvested
Neem	78	10	

3.3 RAW MATERIAL PREPARATION AND OIL EXPELLING

3.3.1 Raw material selection and Cleaning

Seeds should be dried before bagging and storing. Very damp seeds will feel humid when you bury your hand in them, especially if the seeds are warm. When drying the seeds in the sun under a sheet of clear plastic, if the seeds are too wet, moisture will collect beneath the plastic. Sun drying is the easiest way of seed drying.

Moisture percentages of the supplied seeds by Practical Action ranged between 10 – 15% of w/w². If the dried raw material contains about 8 - 9% moisture content it is favourable for expelling purposes. Thus, the percentage of spoiled seeds in the sample supplied was high due to high

² w/w - ' by weight' - Please refer glossary for a more complete definition.

moisture content. Further moisture reduction was required. Seeds were spread and dried for sometime in the sunlight. Depending on the climate and the season sun drying may be sufficient. Dried material tends to absorb moisture from the atmosphere. Therefore just prior to the expelling it is advisable to dry the seeds slightly.

When removing the foreign materials from the seeds, light foreign material and dust were removed by blowing or using flat screens with a mesh. The mesh of the screen used for this purpose depends on the size of seeds and impurities. Smaller impurities fall through the screen while the seeds remain on the screen. Large pieces of foreign material were removed by hand.

3.3.2 De-hulling

Depending on the hardness of the shell, a pre-processing such as de-hulling or dehusking is required before the extraction of oil. De-hulling can be done in a roller mill (steel or sand-filled rollers), or even manually. At the NERDC we used a rubber roll sheller for seed de-hulling with small adjustments.

3.3.3 Expelling methods

Oil can be extracted using the following methods:

- Wooden/stone sekku (mortar and pestle, which is one of the traditional methods)
- Batch type oil press
- Continuous expeller

Presently the edible oil is extracted through traditional *chhque* and *pahha* methods. The oil quantity recovered in the *pahha* method is less and of inferior quality. The capacity is also less when compared to the improved expellers. Oil extraction can be more effectively carried out by the methods described below:

There are batch type presses ranging from small, hand-driven models to power-driven commercial scale hydraulic presses.

The batch press method is used for oil extraction to get high quality oil at a low temperature. The process is also known as the cold press method, and the oil (content) yield recorded is comparatively less than that reported when using a continuous screw expeller.

At the beginning, for experiments both expelling methods (the batch press method and continuous screw expeller) were applied. Oil was extracted from the different types of oil seeds using these different technologies and the most suitable methods and machines for community level oil extraction were identified.

3.4 OIL EXPELLING MACHINES

3.4.1 Batch type hydraulic jack operated oil expeller

The batch Cottage level oil expeller (Photo 16) is operated by a 10 MT Hydraulic Jack. The expeller unit, consists of a hollow cylinder made out of stainless steel, a piston, a handle, a pressure indicator gauge and a mounting frame to house all the above. The amount of crushed seed kernel that the expeller can accommodate per batch is 1.5 - 2 kg. After loading a batch into the cylinder the piston (in the form of a circular disc) - which is fitting quite perfectly with the inner walls of the cylinder with minimum clearance and is attached to an arm - is pressed by a hydraulic jack. This forces the oil to be expelled from the feed through the perforated holes. The oil is then collected for filtration and further processing. After complete expulsion, the hydraulic press is released and the residue cake is removed from the cylinder for further processing. The whole operation takes about 30 - 45 minutes per batch.

3.4.2 Continuous Screw Expeller

The first stage of oil expelling is pre-pressing, using a high pressure continuous screw press called the expeller. Continuous screw expellers have a rotating screw inside a horizontal cylinder (barrel) that is made out of mild steel with 1 - 0.05 mm slots in each 25mm. (The discharge end is open and clearance can be adjusted to suit different kind of raw material.) The screw forces the seeds through the cylinder, gradually increasing the pressure on the crushed seed pulp, slowly moving it to the discharge end. The material gets heated due to friction and the oil escapes from the cylinder through small slots while operating the machine, and pressed residue emerges from the end of the cylinder.



Photo 16 Jack operated oil expeller

First stage of extraction is pre –pressing; that is passing the crushed raw material through the continuous screw expeller in order to preheat the sample. The pressure can be adjusted to suit different kinds of seeds in order to get the highest oil yield. Two or three passes might be required depending on the type of raw material. Extracted oil is filtered, and the material removed from the oil is fed back into the stream (new batch) along with the fresh material. The material which is finally discharged from the pressed residue is called 'pressed cake'(poonack).

The screw type expeller was selected for seed oil expelling at the Gurugoda site.

3.4.3 Reasons for selection of the Continuous Screw Expeller

The capacity of a continuous screw expeller is approximately 4.5 litres/hr. for domba seed oil, 2.5 litres/hr. for rubber seed oil, 2.25 litres/hr. for neem seed oil and 3.00 litres/hr for jatropha seed oil. The batch type hand operated oil expeller's capacity is approximately 2.0 litres/hr for domba seed oil and 0.75 litres/hr for jatropha seed oil. Thus, the continuous screw expeller can be recommend for the project site.

3.4.4 Oil from Pressed Cake

After expelling the seeds, it is necessary to use solvent extraction to get the final traces of oil from the pressed cake. Solvent extraction is a 'high technology' process that has to be carried out on a comparatively large scale. Capital costs are high. Essentially the process is one of continuous counter-current extraction with the raw material flowing in one direction against a solvent; usually hexane. The crude oil then passes on for refining. For the present study it was therefore decided not to extract the oil from the press cake but to use the oil within the press cake for uses such as candles, biogas feed stock etc.

3.5 EXPELLING

3.5.1 Jatropha seeds expelling

Jatropha fruits are mechanically / manually de-hulled. From the fruits the extracted jatropha seeds have a thin hard shell, (Photo 17) which can directly be fed to the continuous expeller available at the NERDC.

The oil content in jatropha seeds (with seed cover) is reported to be in the range of 30 - 50% by weight of the seed and ranges from 45 - 60% weight of the kernel itself.



Photo 17 Jatropha seeds

Jatropha seeds expelling using Continuous Screw Expeller

Jatropha seeds collected from Practical Action were expelled with seed cover. Approximately 16 - 17% w/w oil was recovered. As a mechanical extraction method was only used for oil extraction, the oil content

recorded here was comparatively less than that reported in literature. Some oil might have been lost in the expeller and the seeds were very old. The efficiency of extraction depends on the moisture content of the nuts and the percentage of shell present. The best yields are obtained with 8 - 9% (w/w) moisture. Trial tests were limited, mostly due to a lack of raw materials.

Another sample of fresh seeds (fresher than the earlier batch collected by Practical Action) was collected from near the NERDC premises and manually de-hulled to get kernel. The solvent extraction from this second sample of *Jatropha* seeds kernels gave a 43 - 44% w/w oil yield from the seed sample.



Photo 18 Feeding of *Jatropha* seeds



Photo 19 Expelling of Seeds

3.5.2 Neem Seeds Expelling

Approximately 16 - 17% w/w oil was recovered from neem without removing seed cover. Whole dried seeds directly passed through to the expellers.

A sample of fresh seeds was collected from Dambulla area and tested for total available oil content. The solvent extraction from this sample of neem kernels gave a 38 – 39% w/w oil yields from the seed sample.

The results obtained are higher as the samples are fresher than the samples used earlier. Due to its bitter taste, the pressed cake generated after extraction of the oil from neem seed has no value for animal feed.



Photo 20 Neem seeds

3.5.3 Extraction of Castor Oil

Extraction of oil from castor seeds was done using a similar method as used for most of the other oil seeds. At the beginning direct expelling was done but the yield was very poor. However, oil yield can be increased by steaming and drying the seeds prior to expelling.

Based on literature³, the following method was also tried. The ripe seeds were allowed to dry to enable them to split open naturally. These seeds were then cleaned, cooked and dried prior to extraction. Cooking was done to coagulate protein (which is necessary to permit efficient extraction) and to free the oil for efficient pressing. This method can only be applied to castor seeds (from the seeds studied). The castor seeds collected from Practical Action were expelled with their seed cover. Approximately 17 - 18% w/w oil was recovered from castor seeds.



Photo 21 Castor Seeds

3.5.4 Rubber seeds expelling

All the seeds other than rubber seeds can be expelled with their seed cover. At least partial de-hulling is required before the expelling of rubber seeds. Rubber seeds were collected from Practical Action. De-hulling was done at NERDC using a rubber roll sheller with some small adjustment to the rubber rollers. The rubber roll sheller can be driven by coupling it to a two wheel tractor. Foreign particles were removed and seeds were separated from the shells before oil expelling. A single phase, screw type expeller was used for expelling. The best yields are obtained with 8 - 9% moisture and 15 - 20% shell. The kernel makes up 54 - 60% of the rubber seed weight depending on the moisture content.



Photo 22 Using rubber roll sheller



Photo 23 Rubber kernel makes up 60% of the seed weight

³ Anon. No date. *Principles of Oil Extraction Publishing*. 11.p., available at www.itdgpublishing.org.uk

For rubber seeds the seed cover percentage is comparatively higher than that of the other seed varieties. The study showed that the process of rubber seed extraction could be considerably improved by adding rice husk which improves the friction between particles.

3.5.5 Domba Seeds Expelling (*Calophyllum inophyllum*)

Domba is a non-edible oil bearing plant. The fruit (the ballnut) is round, reaching 2 to 4 cm in diameter and having a single large seed. The seed has a soft shell and is much richer in the quantity of extractable oil than most of the other seeds. The available oil content is 60-64% w/w oil and 50-54% can be extracted.

The nuts should be well dried before cracking, after which the oil-laden kernel should be further dried. The seeds yield a thick, dark green oil for medicinal use or hair grease. This oil is also suitable for biodiesel processing.



Photo 24 Domba Kernal



Photo 25 soft shell

3.5.6 Karanda (*Pongamia pinnata*) seed expelling

Pongamia oil is a non-edible oil extracted from seeds of *Pongamia pinnata*. Each pod bears a single seed. The average fresh weight of a matured seed is 1.2 gm.

Seed oil has many traditional uses as well as being resistant to pests. The oil content is 25 - 30%, however by using the continuous screw type expeller only 5 - 7% w/w oil can be extracted.



Photo 26 Karanda seeds



Photo 27 Karanda seed kernal

Figure 3.1 - Process flow diagram for processing of Rubber and Domba seeds.

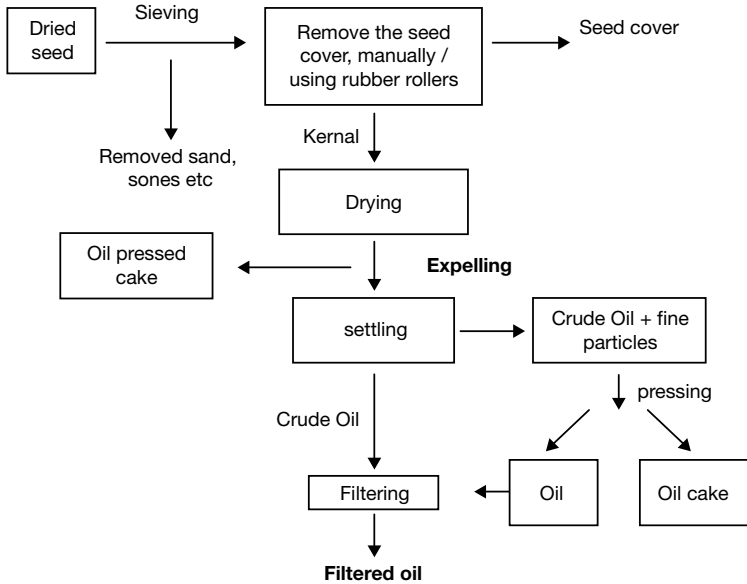
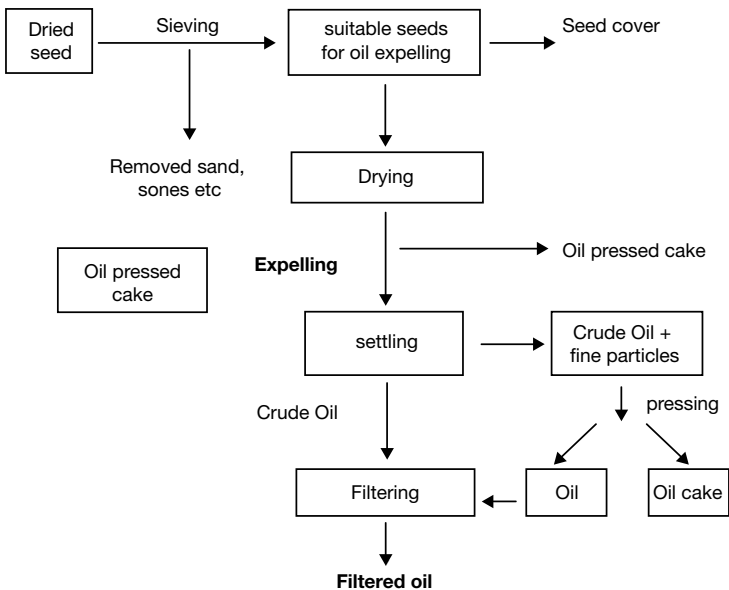


Figure 3.2 - Process flow diagram for processing of Neem and Jatropha seeds

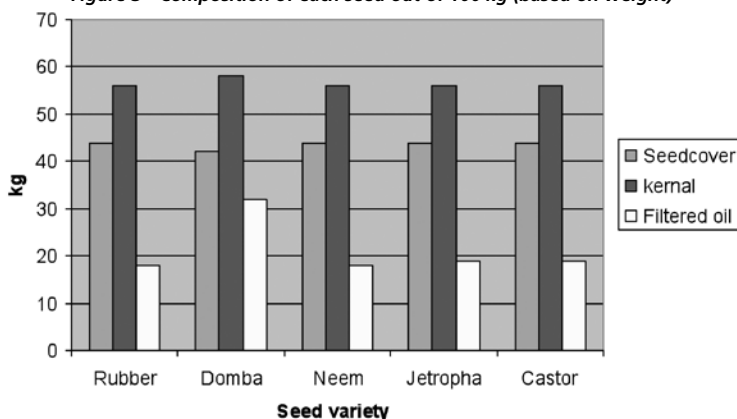


3.5.7 Summary of Seed oil extraction test

Table 3.1 – Seed varieties and weights

Seed variety	Weight..of seed cover / (kg)	Weight of Kernal / (kg)	Weight of oil / (kg)
Rubber	46	54	18
Domba	42	58	32
Neem	44	56	18
Jatropha	44	56	19
Castor	44	56	18

Figure 3 - Composition of each seed out of 100 kg (based on weight)



3.6 OIL CLARIFICATION

After extraction, the oil is clarified to remove contaminants such as fine pulp, water and resins. Raw oil can be clarified by allowing it to stand undisturbed overnight and then removing the upper layer which is called crude oil. The crude oil requires further filtering before processing into biodiesel. After removing the upper layer of the oil, the sedimentation (oil + fine particles) is taken from the bottom part of the container for further separation oil. The sedimentation part requires quick separation because trace amounts of moisture remains in the sludge.

The first step, crude oil was transferred to a Gravity Filter (Photo 28) which was fabricated at the NERDC workshop for fine filtering. The total height of the unit is about 2 meters. Oil filtration takes place under hydrostatic pressure through a 100% polyester filter cloth bag (triple layer, 45 cm height, 8 - 10 cm wide bag which is attached to the lower end of the pipe). After separation of crude oil and sludge, the oil is poured into the Gravity Filter and the filtered oil is immediately taken for the refining process to avoid it getting rancid.



Photo 28
Gravity type oil filter



Photo 29
Fine particles in cloth bag



Photo 30
oil separation using press equipment



Photo 31
oil draining

Further investigations showed that after gravity separation of oil from pulp, the fine particles remaining at the bottom of the cloth bag also need to be taken for oil separation (Photo 29). Therefore as the **second step**, a quick separation process was developed to separate the remaining oil in the sediment from the oil settling container and fine particles in the cloth bag (Photo 29). In this second step, the sedimentation was put in to a suitable type of cloth bag (preferably double layer raw cotton of size 45cm height and 8cm diameter). The bag is then placed in the perforated stainless steel cylinder and pressed by means of a jack operated piston (Photo 30). Due to the resulting pressure exerted into the cloth bag and the built up pressure in the cylinder the oil runs through the pores of the cloth and between the perforated cylinder into the pan or vessel. After the oil has drained (Photo 31), the pressed solid material removed from the bottom part of the cylinder is called oil cake.

3.6.1 Remaining oil in the press cake

Oil in the pressed oil cake, removed from the press equipment was a little bit higher than the oil from the pressed cake (Photo 33) extracted from the continuous type expeller (poonack). Oil pressed cake contains about 15-17% w/w as a remaining oil.

After separation of the oil using the first step and second step, the two end products are filtered oil (ready for processing of biodiesel) and pressed cake (poonack). It is recommended that the pressed oil cakes be fed back into the stream of fresh material (seed) during expelling.



Photo 32 Pressed oil Cake



Photo 33 Pressed cake (Poonack)

3.7 Utilization of pressed cake

The study also tried to focus attention on the uses of pressed cake for producing biogas and to optimise the overall energy efficiency. Subsequent to the removal of the remaining oil in the press cakes, neem pressed cake and rubber pressed cake were already tested for biogas production at a laboratory scale.

For laboratory scale bottle experiments (Photo 34) two varieties of pressed cake (poonack) samples were selected. Before starting the bottle experiments carbon and nitrogen percentage were tested at University of Ruhuna.

Table 3.2 Carbon and Nitrogen in Pressed Cake

Pressed cake	Carbon %	Nitrogen %
Neem	35.7	1.1634
Rubber	35.9	3.45

Pressed cake (3kg in each) has two main uses: as a feeding material for biogas production and as an insecticide (especially neem seed cake).

Subsequent to the removal of the remaining oil in the pressed cake the pressed residues are ready for initial biogas bottle testing.

Raw material - Neem and Rubber residue (3kg in each pressed cake)

Bottle Capacity - 10 litre Aspirator bottle

Biogas was generated using neem but the rubber pressed cake did not give positive results. Gas produced by the neem pressed cake was satisfactory and it burnt with a blue flame but amounts have yet to be quantified.



Photo 34 Press residues are ready for initial biogas bottle testing



Photo 35 Lab scale bio gas unit – 10 litre aspirator bottle capacity, NERDC developed small biogas burner, neem residue and straw raw material

3.8 SOLVENT EXTRACTION

In order to remove the oil from the seed final product pressed cake, it is necessary to use solvent extraction. As mentioned earlier, solvent extraction is a high technology process carried out on a comparatively large scale and having a high capital cost. Due to the large scale required, it would seem unlikely that solvent extraction would find much application in small scale oil seed processing. Solvent extraction was only undertaken in this study to obtain the required details for experimental purposes.

The objective of this research was to determine the available oil in each selected seed variety (Table 3). Solvent extraction is not recommended for community based projects.

Table 3.3 Oil percentages in Seed Varieties

Sample	Oil % (Available oil content in good quality seed kernel)	Oil % (Remaining in pressed cake)	Remarks (5hp, continuous type expeller)
Rubber	32 -36	15-16	Sample after 2nd expelling. (Expeller is not suitable for rubber seed.)
Neem	32- 40	9 - 11	Sample after 2nd expelling (Expeller is suitable for neem seed.)

Domba	64- 68	7-8	Sample- after 3rd expelling (Expeller is suitable for Domba seed.)
Karanda	20- 25	12 -17	Used 5, continous type expeller (Expeller is not suitable for karanda seed)
Jatropha	35- 42	9-11	Sample -after 2nd expelling (Expeller is suitable for jatropha seed)

3.9 RECOMMENDATIONS AND CONCLUSIONS

The remaining oil in pressed cake can also be extracted with solvents, but solvent extraction is a complex operation. Practical Action (formerly known as The Intermediate Technology Development Group), Technical Brief - 'Principles of Oil Extraction', describes several methods of extraction. It cautions that "solvent extraction is not suitable for small-scale processing because of high capital and operating costs, the risk of fire and explosions from solvents, and the complexity of the operation. Waste management would probably also be a problem.

The extraction procedures used have a great influence on the yield of oil, quality of oil and quality of the pressed cake (poonack).The chemical composition of oils from different varieties varies considerably. In addition, the yield of oil generally depends on the storage time, seasonal variation, the sampling methods and the storage condition of the seeds and so on. These variables affected the preliminary studies. These priorities once identified helped to strengthen the quality of existing seeds collected, their transportation, storage facilities, sampling and expelling.

(Further investigations showed that after gravity separation of oil from pulp, the residue remaining in the cloth filter bag used in the oil clarification process still contains oil around 20 - 25% of oil by weight to the fine solid particle)

Part of the oil can be further extracted by steaming the press cake before pressing. This oil content is in economically negligible quantities. However, during the study an attempt were made to extract the maximum yield because this not only increase the yield of oil but also makes it easy for the utilisation of pressed cake with less oil.

Oil pressed cake contains about 15 - 17 w/w % as a remaining oil. It is recommended that the pressed oil cakes be fed back into the stream of fresh material (seed) during expelling.

3.9.1 Recommended Equipment

The screw type expeller was selected for seed oil expelling at Gurugoda

site. Its capacity is about 2.5 litres/hr for rubber kernel oil, 2.25 litres/hr for neem seed oil and 2.25 litres/hr for Jatropha seed oil.

Evaluation of possibilities of seed oil extraction using a commercially available oil expeller was also a part of the study. Although most of the commercially available oil expellers are three phase units, only a single phase power supply is available for communities in Gurugoda. Therefore, 5 HP single phase continuous screw type expeller is suitable for community project purposes.

A continuous screw expeller can be used for coconut oil extraction as well as for other raw materials such as neem, domba and Jatropha. Thus, it enables users not to be restricted to one type of raw material. For this reason the typical continuous screw expeller is recommended for use. The deviation of price and the fabrication of the suggested machine is also favourable when compared to the manually operated batch type presses.

The continuous screw oil expeller has been selected for the biodiesel project for village level implementation because:

- (1) higher production rate (2.5 -5 l/hr)
- (2) continuous operation (instead of batch-type)
- (3) cake produced by the press is in a crushed form ready for application as fertiliser or animal feed without any further grinding or crushing,

The hand-operated press and Gravity Oil Filter (Photo 28) have been selected for oil separation because it can be easily handled by the unskilled community for quick oil separation.



Photo 36 Press
(Fine Particle Remover)

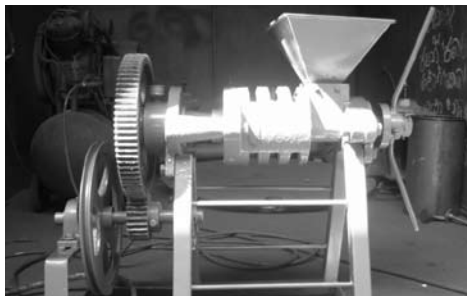


Photo 37 Single Phase Expeller

3.9.2 Training for communities

Before commencing the collection and expelling operations at the Gurugoda site, a team of community members should be formed and the team leader and helpers trained on their roles and responsibilities related to seed expelling. It would be even better if the team were to receive 2-3

days training at NERDC. The results will depend on the experience gained during the training activities.

3.10 CONCLUSIONS

3.10.1 Conclusions from the study

Operational procedures and the working methods have been designed in the following way:

- The oil seeds should be prepared so as to contain low moisture and for there to be a low presence of foreign material (such as sand, stones, wood chippings, etc). These foreign materials can damage the machine, scarring it while in operation when expelling the seeds.
- Seeds having a hard cover have to be de-shelled.
- The de-shelling is to be done by the machine operator and a second person who feeds the machine.
- After being de-shelled/partially de-shelled, kernels should be stored in air -tight bags or containers.
- The storage facilities should be mainly concentrated at the project site because all seeds are seasonal.

3.10.2 Outcomes of the study assessment at the Biofuel Workshop

Ms. Malini Ranatunge made a presentation of the oil extraction study undertaken by her at NERDC. The selected non-edible seeds used for oil extraction were castor, domba, jatropa, rubber and neem. The basic processes involved in oil processing consisted of seed cleaning, seed drying, oil extraction, filtering, packaging and storage of oil. Dehulling was undertaken only for rubber and domba seeds and a rubber roll sheller machine was adjusted to de-hull the seeds. An important factor is the moisture content in the seeds. With moisture it is difficult to expel the oil – therefore storage conditions and drying processes are important.

DISCUSSION:

The following points were voiced during the workshop discussion on the oil expelling study:

- The energy balance needs to be analysed – For small scale plantations chemical extraction may not be suitable and there are also safety issues to be considered.
- Community centre – Other seeds (besides Jatropa) can be used and extraction and collection processes may differ accordingly. When implementing the community based biofuel project an appropriate seed collection centre is vital.
- Seed storage should be discussed – Is filtering and storing suitable or would drying in a solar dryer be more suitable than storage?
- What should also be considered is the fact that rubber seeds release toxic gases.

4

CHAPTER 4

BIODIESEL PROCESSING FROM RAW VEGETABLE OILS

By Dr. C.S.Kalpage, University of Peradeniya

4.1 INTRODUCTION

Biodiesel is a renewable fuel derived from vegetable oil or animal fats that can be added to petroleum diesel as a blend or used on its own in diesel engines. The first diesel engines by Rudolph Diesel in the 1890s were designed to run on refined vegetable oils.

Research into the use of transesterified sunflower oil, and refining it to diesel fuel standards, was initiated in South Africa in 1979. An Austrian company erected the first biodiesel pilot plant in November 1987 and the first industrial-scale plant in April 1989.

The production of biodiesel is now a proven and efficient process. Simplified to its core steps, the process is accomplished by combining refined oil with an alcohol and a catalyst. When the catalyst is removed, the remaining components are biodiesel and a small amount of glycerin. When Practical Action put forward the idea of liquid biofuels, there was no reported research to be conducted on biodiesel processing in Sri Lanka. With the aim of erecting a community level biodiesel processing centre, the TAB led by Practical Action appointed Dr. C.S. Kalpage, a member of the TAB, to conduct biodiesel processing studies at the Chemical and Process Engineering (CPE) laboratory at the University of Peradeniya.

The production of fuel-quality biodiesel from various feedstocks - namely coconut, castor, jatropha, rubber, neem and domba oils - were investigated in the present study. The oils extracted at NERDC were used in the testing. Testing took place at the CPE laboratory at the University of Peradeniya. Dr. Kalpage and his students worked on identifying different methods of processing as well as analysing the main factors affecting the conversion efficiency of the process, such as the molar ratio of CH_3OH to oil, amount of catalyst (both acid and alkali), reaction temperature, time and post-processing. Transesterified oils produced in this study were sent to the University of Ruhuna (ROH) for testing on engine running (Chapter 5).

4.1.1 Precursors

Biodiesel can be produced from a variety of renewable lipid sources such as vegetable oils of soybeans (*Glycine max*), canola (*Brassica napus*), oil palm (*Elaeis guineensis*), peanut (*Arachis glabrata*), olive (*Olea europaea*), corn (*Zea mays*), sunflower (*Helianthus annuus*), etc, and animal fat.

However, some of the oil sources mentioned above are commodities whose prices are strongly dependent on the international market. The food industry also competes directly for these feedstocks - a critical consideration for a world whose population is increasing exponentially. For these and other reasons, non-edible oil sources are preferred for biodiesel production, particularly those requiring low agronomic demand for cultivation, a reasonable plant cycle, favorable geographic adaptability, high oil content, low cost for cultivation and harvesting. However, some technological parameters are also critical, such as the ease with which the oil can be extracted from the oil seed, the susceptibility of transesterification, the fuel properties of the biodiesel derived and the nutritional value of the residual oil cake.

In the context of Practical Action objectives *Jatropha* (*Jatropha curcas*), castor bean (*Ricinus communis*), rubber (*Hevea brasiliensis*), neem (*Azadirachta indica*), domba (*Calophyllum inophyllum*), passion fruit (*Passiflora edulis*), and mee (*Madhuka neriifolia*), are recognised as suitable oils for biodiesel processing.

Oil Chemistry

Vegetable fats and oils are substances derived from plants that are composed mainly of triglycerides. Triglycerides are esters of glycerin and varying blends of fatty acids. Vegetable oils also encompass molecules such as fatty acids and their derivatives including tri-, di-, and monoglycerides and phospholipids. Phospholipids have a polar "head" which is hydrophilic, and a hydrophobic tail composed of fatty acids. However, the presence of phospholipids in oils interferes with the processing as well as the quality of biodiesel.

The chemical formula of tri-glyceride can be represented by $RCOO-CH_2CH(-OOCR')CH_2-OOCR''$, where R, R', and R'' are longer alkyl chains. The three fatty acids RCOOH, R'COOH and R''COOH can all be different, all the same, or only two the same. Chain lengths of the fatty acids in naturally occurring triglycerides can be of varying lengths but 16, 18 and 20 carbons are the most common. Natural fatty acids found in plants and animals are typically composed only of even numbers of carbon atoms due to the way they are bio-synthesised. Bacteria, however, possess the ability to synthesise odd- and branched-chain fatty acids. Consequently, ruminant animal fat contains odd numbered fatty acids, such as 15, due to the action of bacteria in the rumen.

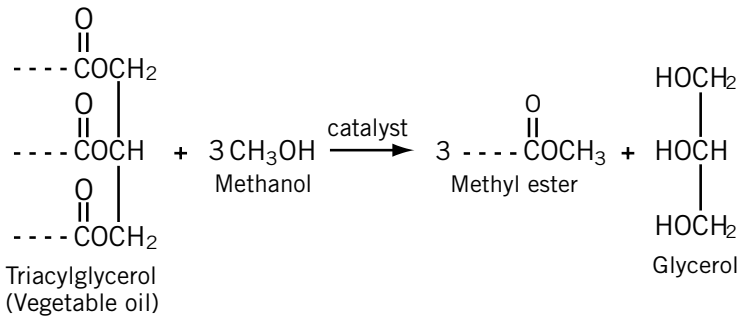
4.1.2 Chemical Reactions

Vegetable oil can be used as diesel fuel just as it is, without being converted to biodiesel. However, straight vegetable oil (SVO) is much

more viscous than the conventional diesel fuel, and it doesn't burn the same in the engine. Many studies have found that the use of SVO can damage existing engines if used without modification.

Two main methods have been developed to make biodiesel from vegetable oils; base catalysed transesterification and acid catalysed transesterification. The most widely adopted process consists of a chemical reaction in which the fat or oil triglyceride (also known as tri-acyl-glycerol: TAG) react with an alcohol (methanol or ethanol) using an alkaline catalyst (usually NaOH or KOH) to produce simple alkyl monoesters (known as biodiesel) and glycerin. Figure 4.1 shows the transesterification reaction of TAG with methanol, yielding methyl esters and glycerol.

Figure 4.1 The transesterification reaction of TAG with methanol



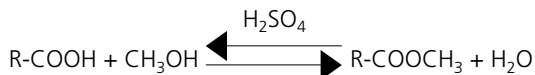
Any free fatty acid in oil will be converted to soap by the process of saponification in the base catalysed process (Figure 4.2). Soap will interfere with glycerin separation and produce a low quality biofuel. However, base catalysed reactions are faster than the acid catalysed reactions.

Figure 4.2 Free fatty acid reaction with NaOH



In addition to products shown in Figure 4.1, acid catalysed transesterification produce monoalkyl esters from free fatty acid. Figure 4.3 shows the chemical reaction of acid catalysed esterification of fatty acids. The overall reaction takes place in three steps - each step in the process is reversible. The esterification step yields a water molecule as a by-product. However, in the presence of a large amount of excess alcohol, the equilibrium point is displaced and the transesterification can be virtually completed.

Figure 4.3 Acid catalysed esterification of fatty acids



Thus, acid catalysts transesterification results in very high yields of alkyl esters, but the reaction rate is slower when compared to base catalysed transesterification. The alcohol to oil molar ratio is one of the main factors that influence the transesterification. An excess of the alcohol favours the formation of the products.

4.1.3 Products and By-Products

Biodiesel that is prepared by transesterification should be post-treated to comply with an international standard for safe use in engines. Engine manufacturers and biodiesel plants in different parts of the world use slightly different standards (see Annexure 4). Virtually all modern diesel engine warranties permit the use of biodiesel that meet a specification prescribed by the manufacturer.

Glycerol is the main by-product of the esterification (see the reaction in Figure 4.1). It is typically recovered by gravity settling or centrifugation. Refined glycerol can be used in many industrial applications.

4.2 STUDIES CONDUCTED AT THE CPE LABORATORY

4.2.1 Materials

Given below in Table 4.1 is a list of raw oils received from NERDC for processing studies

Table 4.1. Raw oils supplied by NERDC

Received on	Type	Quantity / (L)
11/07/2007	Castor	4.50
15/08/2007	Castor	10.00
	Refined Castor	0.42
	Jatropha	5.00
	Refined Jatropha	0.25
	Virgin coconut oil	0.25
19/10/2007	Rubber	10.0
	Refined Rubber	2.00
	Refined coconut oil	0.25
03/12/2007	Coconut	13.00
21/01/2008	Neem	2.00
	Domba	0.75
29/02/2008	Neem	6.00
	Rubber	10.0

Other chemicals such as methanol, sodium hydroxide, isopropyl, sulphuric acid, phenolpalene, etc, were purchased from various chemical suppliers. They were all commercial grade chemicals.

4.2.2 Methods

Descriptions of the one-step and two-step methods, methanol test, wash test, and biodiesel washing can be found in Annexure 1. These include details of the reagents, apparatus and procedure used for these methods.

4.2.3 Results

a) Castor Oil Processing

All tests with castor oil failed to make biodiesel in the one-step trials. These tests resulted in jelled solids (see photo 38) or liquids which failed in the washing test. This result was attributed to the high acid or phospholipid contents of the oil.

Oil refining by various methods of soap formation and separation, through the use of different concentrations of aqueous NaOH solutions, different masses of NaOH flakes, sodium meta-bisulphite (SMS), etc., were tested. Unfortunately, all these methods yielded significantly less amounts of refined product. Aqueous NaOH solutions reduced of the free fatty acid (FFA) Value of raw castor oil from 6.5 to 1.5%, but with a reduction of 75% of the initial volume due to soap formation. The maximum acid value reduction by SMS was only from 6.5 to 4.2%.

Oil refining by acid esterification started in September 2007. Sulphuric acid (H_2SO_4) was used as the catalyst in the esterification step. Different ratios of methanol (CH_3OH) and acid were used. Final FFA contents for oil:acid:methanol volume ratio of 100:1:10, 100:1:20 and 100:1:30 were 4.8, 2.5, and 1.8% respectively.

In the 2nd step (transesterification), most studies resulted in a transparent solution, but any phase separation was hardly achieved. The single phase



Photo 38: Reaction of castor oil with $NaOCH_3$ resulted in a jelled solid

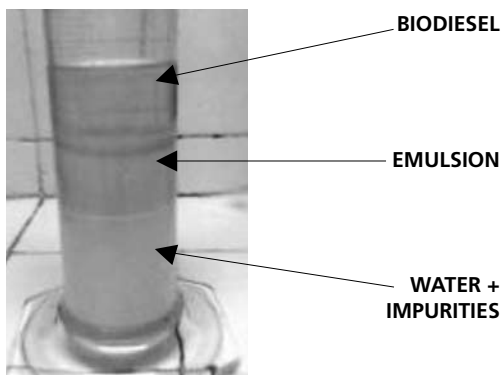


Photo 39 Wash Test result of CaME after two-step Process by slightly acidic water

indicated that either the glycerine was not formed at all or a very little amount was formed and miscible in the biodiesel (castor oil methyl ester - CaME) fraction. Due to the lack of analytical equipment such as a gas chromatograph, this could not be verified.

Rigorous shake in the subsequent washing step yielded an emulsion layer. However, when washing was performed by sprinkling water from top, a little amount of biodiesel was separated (approximately 50 ml of refined biodiesel from 200 ml of raw oil after 5 days of settling). Later it was found that when the crude biodiesel from castor oil was bubbled with slightly acidic water by the method of "Idaho University Bubble Wash Technique", a reasonable amount of cleaned biodiesel can be separated.

b) Jatropha Oil Processing

Both one-step and two-step biodiesel procedures with various chemical ratios were tested for jatropha oil samples. The one-step method resulted in jelled product in all tests similar to the result described in Section 4.2.3 a). However, the two-step procedure was successful for jatropha. The FFA of jatropha was reduced from around 11.5 to 3.8% in the first step of the two-step method and to below 0.4% in the subsequent transesterification. Though water was readily separated in the wash test, the top layer was not transparent enough to be considered as pure jatropha methyl ester as reported in biodiesel literature.

c) Coconut Oil Processing

Small batches of coconut oil biodiesel (coconut oil methyl ester - CME) were successfully produced from locally purchased coconut oil using the one-step method. Later, Practical Action supplied approximately 15L of pure coconut oil to make CME and test in an engine at the University of Ruhuna. Initially 2L of oil was transesterified using the one-step method. A few seconds after the reactants were added (methanol/sodium hydroxide mixture into the heated oil), the whole stock solidified (Photo 40).



Photo 40 Solidified product in the CME processing

It was found later that the acid value of the supplied coconut oil was extremely high; (6 ml NaOH/ml oil \approx 4.7% FFA). Therefore, these tests with coconut oil helped to determine that the acid value as the main parameter that would drive the saponification (soap making) reaction instead of the transesterification reaction. Subsequently, the two-step procedure was tested and satisfactory results were obtained. Thereafter, the supplied coconut oil stock was transesterified and 5 L each of unrefined and unrefined CME was sent to the University of Ruhuna.

d) Rubber Oil Processing

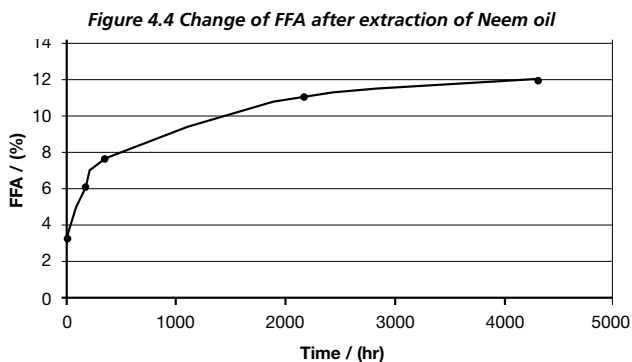
Biodiesel trials with rubber seed oil started in October 2007, but without much success until early December. As similar to previous observations with jatropha, castor and coconut oil the one-step method totally failed. This was attributed to the high FFA content of the rubber seed oil.

FFA reduction by esterification with various reactant ratios was tested. Altogether 24 different systems were analysed. Given in Annexure 2 is an extended abstract published at the Peradeniya University Research Sessions (PURSE 2008) on the studies with rubber seed oil.

e) Neem Oil Processing

On 21st January 2008, a small amount of neem oil was extracted on a test study conducted at Udaya Industries, Geliyoa. Acid values were determined as soon as the samples were received and continued to monitor for about six months. Figure 4.4 shows the percentage of FFA versus time after extraction of neem oil. The result indicated that the acid value of oil increases rapidly after extraction and the rate of change dies out as time passes. Therefore it can be recommended to process vegetable oil as soon as they are extracted.

Transesterification experiments by two-step method were conducted with oil supplied by Practical Action. A complete study was performed. Results were published at the PURSE 2008 (see Annexure 3)



f) Domba Oil Processing

A small amount of domba oil was also extracted together with neem oil at the expeller testing at Udaya Industries. The one-step transesterification procedure was not successful but the two-step method produced satisfactory results as similar to all previous studies.



Photo 41 Biodiesel samples processed at the CPE laboratory (from left to right: jatropha, castor, rubber, neem, domba and coconut)

4.3 SAFETY ISSUES

Some chemicals used in biodiesel processing are extremely harmful. Therefore, proper safety measures must be followed. It is extremely essential to wear safety glasses, masks, coats, shoes and chemical resistant gloves when handling highly toxic chemicals like methanol and concentrated sulphuric acid. Remove any source of ignition (lighters, hot plates, hot air guns, etc.) from the working area. Ensure that the work area is very well ventilated.

If chemicals accidentally come into contact with the eyes or skin, immediately flush with plenty of water. Continue for several minutes and call for medical help. If clothes are soaked with toxic chemicals, immediately remove any contaminated clothing and store well away from a source of ignition (preferably outside).

In case of disposal, trace amounts can be flushed down a sink with a large quantity of water, unless local rules prohibit this. Larger amounts should be collected in a container for disposal. (Further safety information such as material safety data sheets for methanol, ethanol, sodium hydroxide, sulphuric acid, phenolphthalein and sodium methoxide are available on the internet.)

It is very important to employ trained staff members in biodiesel processing activities.

4.4 CONCLUSION AND RECOMMENDATIONS

4.4.1 Conclusions from the study

Fuel grade biodiesel can be produced by transesterification; a reaction of

oil with a primary alcohol in presence of a catalyst. Methanol or ethanol is used as the primary alcohol. Suitable catalysts are of two types: acid (for example sulphuric or hydrochloric) or alkali (sodium or potassium hydroxide). Reaction rate with alkali based catalyst are fast but not suitable for all types of oil. Acid based transesterification was observed as being an extremely slow process. The main criteria that will determine the type of catalyst is the free fatty acid (FFA) content of the respective oil. When the FFA level is less than about 1.5%, its effect can be ignored and transesterification can be performed in one step (the one-step method). This method is simple, low cost and easy to perform. In general edible oils contain a lower amount of FFA, therefore they can easily be converted to biodiesel using the one-step method.

Theoretically, the transesterification reaction yields three molecules of respective monoesters and one molecule of glycerol from each molecule of triglyceride. Monoesters (i.e. biodiesel) and glycerol have different densities; therefore, can easily be separated by gravity settling.

Production of fuel-quality biodiesel from various feedstocks - namely coconut, castor, jatropha, rubber, neem and domba oils - were investigated in this study. Unfortunately all non-edible oils considered in this study reported high FFA values (jatropha, 16%; castor, 16%; rubber, 28%; neem, 14% and domba, 21%). Transesterification of these oils with alkali based catalyst yield a considerable amount of soap. Soaps are emulsifiers that make the separation of the glycerol and ester phases less sharp. Soap formation also produces water that hydrolyzes the triglycerides and contributes to the formation of more and more soap. Further, the catalyst that has been converted to soap is no longer available to accelerate the biodiesel forming reaction therefore high catalyst loading is required.

Oil pre-treatment studies were conducted when the oil FFA levels were high (< 4%). This allowed the catalyst to neutralise the FFA by forming soap. However, due to the presence of an excessively high amount of FFA, a reasonable quantity of oil could not be recovered.

Acid catalysed transesterification was found to be a good solution to prevent soap formation. However the reaction rate was considerably less, requiring lengthy reaction periods. Therefore a two-step method was adopted; acid catalysed reaction followed by alkali catalysed reaction.

The main factors affecting the conversion efficiency of the process, such as the molar ratio of CH_3OH to oil, amount of catalyst (acid in esterification and alkali in transesterification), reaction temperature and reaction duration, were studied. It was found that an excess of the alcohol favours the formation of the products. However, an excessive amount of alcohol made the recovery of the glycerine difficult, so the ideal alcohol/oil ratio

has to be established empirically for each of oils, considering each individual process. The maximum reaction rates were observed when the temperature was above 45°C.

When the first step was manipulated to reduce the FFA content below 1.5%, the second step satisfactorily produced biodiesel. Process optimisation studies were conducted with varying chemical composition, reaction time, and temperature.

4.4.2 Recommendations

1. When a feedstock is received store it in an air tight container. Transesterify the oil as soon as possible.
2. Before proceeding to process large scale batches, always start with lab-scale (200 ml) batches.
3. Measure the acid value and choose the appropriate transesterification procedure.
4. Ideal alcohol/oil ratio has to be established empirically.
5. Conduct a methanol test and a wash test prior to biodiesel refining.
6. Wash and purify biodiesel only if it is recommended by University of Ruhuna after engine tests.

4.4.3 Outcomes of study assessment at the Biofuel Workshop

Dr. Kalpage presented his methodology and findings to the TAB at the Biofuel Workshop. He highlighted the difference between the one-step and two-step method used – with the former only used for oils extracts of acid value 3.5 and below. Most of the oils used in the study had an acid value of over 3.5. Therefore the two-step method was used.

Discussion

- Further testing of bio-diesel produced is required to ascertain if they meet bio-diesel specifications
- Glycerine and the potential for it to be made at the commercial level need to be studied (however the Glycerine needs to be purified in order to be used and sold).
- It was highlighted that safety is a priority – especially in a community environment

5

CHAPTER 5

TESTING OF TRANSESTERIFIED JATROPHA OIL

By Dr. H. C. Ambawatte, University of Ruhuna

5.1 INTRODUCTION

Some development work has been carried out with regard to the production of transesterified non-edible oil and its use in biodiesel, by units such as the Indian Institute of Science, Bangalore, the Tamil Nadu Agriculture University Coimbatore and Kumaraguru College of Technology in association with Pan horti-consultants, Coimbatore. Generally a blend of 5% to 20% of biodiesel is used in India (B5 to B20). The Indian Oil Corporation has taken up research and development (R&D) work to establish the parameters of the production of transesterified Jatropa Vegetable oil and the use of biodiesel in its R&D center at Faridabad. Research is carried out in the Kumaraguru College of Technology for marginally altering the engine parameters to suit the Indian Jatropa seeds and to minimise the cost of transesterification.

In this study transesterified oil received from the research conducted at the University of Peradeniya was applied to diesel engines. Testing was carried out by Dr. H. C. Ambawatte and his team at the Chemistry Department and the Mechanical and Manufacturing Engineering Department (DMME) of the University of Ruhuna. Testing was done using a diesel engine fitted with a generator which generated electricity. This was used to measure electrical loads. The jatropa oil properties were tested and different blends of jatropa oil with diesel were also tested in a diesel engine. For the latter process 4 experiments were conducted involving diesel fuel and different blends of jatropa oil and diesel fuel.

5.2 TESTING OF TRANSESTERIFIED JATROPHA OIL PROPERTIES

The transesterified jatropa oil sample was tested to determine its following properties; density, specific gravity, viscosity, and flash point. Table 5.1 represents the results of such tests. The importance of the above properties are described below.

Density

The greater the fuel density, the greater the mass of fuel that can be stored in a given tank. Thus, the greater the mass of fuel than can be pumped for a given pump. Fuel density generally increases with increasing molecular weight of the fuel molecules. Fuel density also generally increases with increasing molecular weight of the component atoms of

the fuel molecules. Fuel density is used to calculate fuel volume ratio, which is in turn used to calculate the tank mass.

Specific Gravity

In relationship to liquids, the term specific gravity is used to describe the weight or density of a liquid compared to an equal volume of fresh water at 4°C (39°F). Specific gravity helps calculate its density.

Viscosity

The viscosity of a fluid is its resistance to shear or flow, and is a measure of the fluid's adhesive/cohesive or frictional properties. The viscosity will arise due to internal molecular friction within a fluid producing the frictional drag effect. It is a very important property when the substitution of fuels is considered. Preferably the viscosity of biofuel or fuel blend should be similar to that of diesel oil.

Flash Point

The flash point of a chemical / fuel is the lowest temperature where enough fluid can evaporate to form a combustible concentration of gas. The flash point is an indication of how easily a chemical may burn. Materials with higher flash points are less flammable or hazardous than chemicals with lower flash points. It is very important to have the flash point of biodiesel or fuel blend similar to or less than that of diesel fuel in order to have proper combustion of fuel supplied to the cylinder.

Table 5.1: Properties of Transesterified Jatropha oil sample

Properties	Unit	Transesterified Sample	Standard value for Biodiesel (ASTM D 6751)
Density	g/cm ³	0.9037	0.87 - 0.89
Specific Gravity	-	0.9074	0.88 - 0.90
Viscosity	mm ² /s	22.0	1.9 - 6.0
Flash point	°C	39.3	100 - 170

The analysis of these results is given in Section 5.4.1.

5.3 TESTING THE PERFORMANCE OF JATROPHA OIL BLENDS IN A DIESEL ENGINE

Results are based on tests undertaken on a single-cylinder direct-injection engine operating on diesel fuel, blends of diesel and jatropha oil (in the proportion of 95%:5%), and blends of diesel and jatropha oil based biodiesel (in the proportions of 95%:5% (B5) and 90%:10% (B10)). The results covered a range of operating loads from 0 to 7 kW on the engine. Values are also given for the specific fuel consumption. The test showed

that jatropha oil based biodiesel as well as blends of jatropha oil with diesel oil could be conveniently used as a diesel substitute in a diesel engine.

Table 5.2 Technical Details of the Engine

Make	Yanmar Diesel Engine, Japan
Type	Single cylinder, Direct Injection, CI engine
Continuous Output	6.3 kW @ 2400 rpm
Maximum Output	7 kW @ 2400 rpm
Displacement	0.450 l
Loading device	Electrical dummy load

The test further showed a reduction of brake specific fuel consumption for jatropha oil and its blends with diesel. When starting the engine the specific fuel consumption is lower for jatropha oil and its blends with diesel when compared to diesel. Thus, this results in better engine performance than diesel fuel, suggesting that jatropha oil can be used as an ignition-accelerator additive for diesel fuel.

5.3.1 Experiment 01:

Firstly the test was done for the diesel fuel. The measurements were taken increasing load from 0 W to engine maximum load 7 kW by an increment of 500 W and decreasing load from 7 kW to 0 W by the same increment. The observations are given in Table 5.3.

Table 5.3 Observation for Diesel Fuel

Load (W)	Acceleration Time / (s)		Fuel Consumption / (ml)	
	When Increasing Load	When Decreasing Load	When Increasing Load	When Decreasing Load
0	85	58	15	10
500	70	50	15	10
1000	42	43	10	10
1500	35	35	10	10
2000	50	31	15	10
2500	25	25	10	10
3000	23	21	10	10
3500	32	12	15	5
4000	23	20	10	10
4500	18	17	10	10
5000	26	31	15	20
5500	24	24	15	15
6000	15	15	10	10
6300	30	8	20	5
6500	15	15	10	10
7000	18	12	15	10

Density of Diesel Fuel sample = 0.850 kg/m³

Table 5.4 Calculation for Diesel Fuel

Load (kW)	Brake Specific Fuel Consumption (BSFC) (kg/kWh)
0	-
0.5	1.275
1.0	0.720
1.5	0.583
2.0	0.472
2.5	0.490
3.0	0.464
3.5	0.397
4.0	0.356
4.5	0.389
5.0	0.376
5.5	0.348
6.0	0.340
6.3	0.320
6.5	0.314
7.0	0.364

5.3.2 Experiment 02:

Secondly the same test was done for the blends of diesel and jatropha oil based biodiesel in proportions of 95%:5% (B5). Observations are given in Table 5.5.

Table 5.5 Observation for B5

Load (W)	Acceleration Time / (s)		Fuel Consumption / (ml)	
	When Increasing Load	When Decreasing Load	When Increasing Load	When Decreasing Load
0	22	21	05	05
500	25	22	05	05
1000	23	19	05	05
1500	37	20	10	05
2000	33	31	10	10
2500	31	30	10	10
3000	27	25	10	10
3500	23	22	10	10
4000	21	20	10	10
4500	18	18	10	10
5000	17	31	10	20
5500	15	15	10	10
6000	23	15	15	10
6300	31	25	20	20
6500	23	14	15	10
7000	22	07	15	05

Density of B5 Fuel sample = 0.8527 kg/m³

Table 5.6: Calculation for B5

Load (kW)	Brake Specific Fuel Consumption (BSFC) (kg/kWh)
0	-
0.5	1.306
1.0	0.731
1.5	0.539
2.0	0.480
2.5	0.403
3.0	0.394
3.5	0.390
4.0	0.374
4.5	0.379
5.0	0.384
5.5	0.372
6.0	0.337
6.3	0.348
6.5	0.319
7.0	0.302

5.3.3 Experiment 03:

The same test was done for the blends of diesel and jatropha oil based biodiesel in the proportion of 90%:10% (B10). Observations are given in Table 5.7.

Table 5.7 Observation for B10

Load (W)	Acceleration Time / (s)		Fuel Consumption / (ml)	
	When Increasing Load	When Decreasing Load	When Increasing Load	When Decreasing Load
0	32	26	05	05
500	21	28	05	05
1000	26	21	05	05
1500	18	18	05	05
2000	16	18	05	05
2500	16	18	05	05
3000	15	17	05	05
3500	28	19	10	10
4000	24	10	10	05
4500	19	17	10	10
5000	16	16	10	10
5500	16	16	10	10
6000	15	16	10	10
6300	29	15	20	10
6500	14	13	10	10
7000	13	11	10	10

Density of B10 Fuel sample = 0.8554 kg/m³

Table 5.7: Calculation for B10

Load (kW)	Brake Specific Fuel Consumption (BSFC) (kg/kWh)
0	-
0.5	1.257
1.0	0.655
1.5	0.570
2.0	0.453
2.5	0.362
3.0	0.321
3.5	0.374
4.0	0.340
4.5	0.380
5.0	0.385
5.5	0.350
6.0	0.331
6.3	0.333
6.5	0.351
7.0	0.367

5.3.4 Experiment 04:

The same test was done for the blends of diesel and jatropha oil in proportions of 95%:5%. Observations are given in Table 5.8.

Table 5.8: Observation for Blends of Diesel and Jatropha Oil

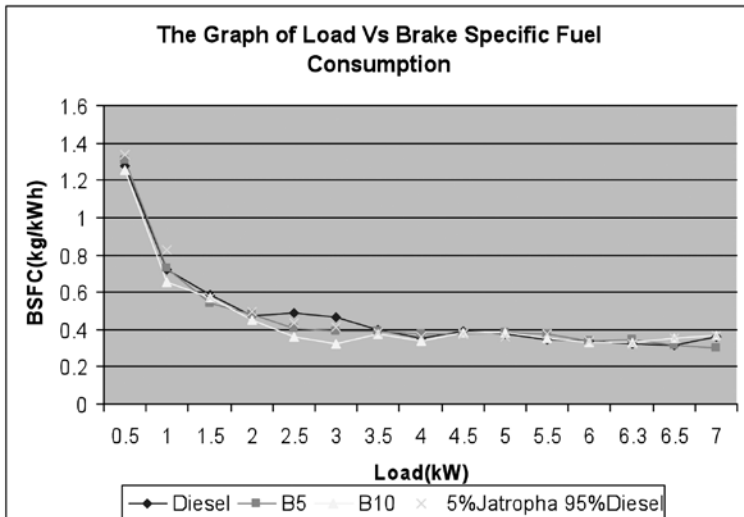
Load (W)	Acceleration Time / (s)		Fuel Consumption / (ml)	
	When Increasing Load	When Decreasing Load	When Increasing Load	When Decreasing Load
0	27	32	05	05
500	22	24	05	05
1000	17	20	05	05
1500	17	19	05	05
2000	13	18	05	05
2500	13	15	05	05
3000	12	13	05	05
3500	11	12	05	05
4000	32	11	15	05
4500	09	18	05	10
5000	17	69	10	40
5500	15	15	10	10
6000	16	14	10	10
6300	16	14	10	10
6500	14	13	10	10
7000	13	11	10	10

Density of blends of diesel and jatropha oil sample = 0.853 kg/m³

Table 5.9: Calculation for blends of diesel and Jatropha oil

Load (kW)	Brake Specific Fuel Consumption (BSFC) (kg/kWh)
0	-
0.5	1.335
1.0	0.830
1.5	0.569
2.0	0.495
2.5	0.439
3.0	0.409
3.5	0.381
4.0	0.357
4.5	0.379
5.0	0.357
5.5	0.372
6.0	0.341
6.3	0.325
6.5	0.350
7.0	0.366

Fig 5.1: Fuel Consumption against Load



5.4 CONCLUSIONS

5.4.1 Conclusions from the study

An experimental investigation was conducted to explore the performance of Jatropha oil in Sri Lanka, comparing its properties with the jatropha oil

in India. The transesterification process and the observations and results obtained suggest the following conclusions.

The important chemical and physical properties of *Jatropha Curcas* oil were determined by standard methods and compared with diesel. The analytical results are shown in Table 5.10. The results show that the density of the jatropha oil is comparable to the diesel oil and is slightly higher than the diesel fuel. However, the kinematics viscosity and the flash point of *Jatropha Curcas* oil are several times higher than those of the diesel oil.

Table 5.10 Comparison of Jatropha Oil Properties

Properties	Units	Experimental Value	Indian Standard Value	Diesel
Density	g/cm ³	0.9098	0.9200	0.836-0.850
Specific Gravity	-	0.9135	0.9186	0.839-0.853
Viscosity	mm ² /s	29.8	40-50	4-8
Saponification Value	-	230.7	195.0	-
Acid Value	-	3.64	3 - 38	-
Flash Point	°C	170.5	240/110	45-60
Calorific Value	MJ/kg	39.3	41.73	42.57

1. The density and specific gravity of the jatropha oil in Sri Lanka are **0.9098 g/cm³** and **0.9135 g/cm³** respectively and the Indian values are **0.9200 g/cm³** and **0.9186 g/cm³** respectively. So the density, specific gravity of jatropha oil in Sri Lanka is slightly lower than those values in Indian standard.
2. The remarkable observation is that the viscosity of jatropha oil in Sri Lanka is very much lower than the Indian value.
3. The saponification value was observed to be much larger than the Indian value. Thus, more moles of potassium hydroxide (KOH) were used for 1 gram of fat making it have more moles in the fat. Therefore the chain lengths are smaller, due to the equation:

$$\text{Number of moles} = \text{mass of oil} / \text{relative atomic mass}$$

The relative atomic mass would be smaller with smaller fatty acid chains, meaning more moles. So the higher saponification value the smaller the chain length.

With regard to findings of properties such as density and viscosity it is favourable to use the jatropha oil in engines in Sri Lanka since these properties are similar to that of diesel fuel. But considering the high saponification value of jatropha oil it is suggested that further esterification and cleaning is required before using jatropha oil based biodiesel in engines.

4. In chemistry, acid value is the mass of potassium hydroxide (KOH) in milligrams that is required to neutralise one gram of chemical substance. The acid number is a measure of the amount of carboxylic acid groups in a chemical compound such as a fatty acid.

The acid number is used to quantify the amount of acid present in a sample of biodiesel. It is the quantity of base, expressed in milligrams of KOH that is required to neutralise the acidic constituents in 1 g of sample.

The experimental acid value is 3.64. But acid values (mg KOH/g oil) for bio-diesel is preferred to be lower than 3.

5. In the transesterification process, two different layers were formed - one which was at the bottom in the separator funnel and was light yellow in colour and the upper layer colour was dark yellow which can be considered as soap formed during transesterification.
6. An experimental investigation was conducted to explore the performance of jatropha oil and its fuel blends with diesel in a direct-injection single-cylinder diesel engine. The results obtained suggest the following conclusions:
 - The main aim of the present investigation was to reduce the viscosity of *Jatropha Curcus* oil close to that of conventional diesel fuel to make it suitable for use in a compression ignition engine and to evaluate the performance of the engine with the modified oils. A significant reduction in viscosity was achieved by dilution of jatropha oil with diesel in varying proportion.
 - Pure jatropha, pure diesel and blends of jatropha and diesel oil exhibited similar performance under comparable operating conditions.
 - The jatropha oil can be used as an ignition-accelerator additive for poor diesel fuels.
 - The jatropha oil has substantial prospects as a long-term substitute for diesel fuels. The B5, B10 fuel blends competed favourably with diesel fuel and offers a reasonable, if not better, substitute for pure diesel.
 - It has been established that *Jatropha Curcas* oil can be substituted for diesel for use in a compression ignition engine without major

operational difficulties. However, the proportion of the blends maybe further improved to make use of a higher percentage of jatropha oil in the blend using a purer grade of jatropha oil which may be obtained through pretreatment of the oil. Moreover, the long term durability of the engine using biodiesel as fuel requires further study.

Future Plans

1. Chemical Analysis of Jatropha oil
The amount of free fatty acids in the oil, types of acids available in oil, iodine value, etc. are required to be tested.
2. Further Testing of properties of Jatropha oil
Cetane values, Pour Point, Iodine Value etc. should be tested.
3. Testing of Oil after Transesterification
Acid value, Cetane values, Calorific value, Water & Sediment, Carbon Residue, Sulphur, Free glycerin and etc.
4. Further testing the performance of Jatropha oil blends in a diesel engine.
Find out the best proportion of jatropha oil blend with diesel for CI engines and their emissions.
5. Research should be done to explore possibilities of using raw jatropha oil in diesel engines by doing engine modification such as selection of a suitable injector pump and injector nozzles etc.

5.4.2 Outcomes of study assessment at the Biofuel Workshop

Dr. Ambawatte presented the findings of his study to TAB at the workshop. He stated that getting adequate oil for testing was difficult and that future testing needs to take place for longer periods of time (in practice) and should not take place in the laboratory. He also stated that technologies dealing with Renewable Energy fuels may have problems in Sri Lanka with regard to warranties (engine and vehicle), registration and insurance. In terms of small scale technologies the above should not be a problem.

Dr. Ambawatte stated that fuel distribution, storage and blending and transportation will be complicated with the existing government policies. In addition, raw vegetable oil (straight vegetable oil) could be used with engine modifications (i.e. modification of fuel supply system, replacing rubber hoses, etc). Hence it is suitable to use a blend of SVO with diesel oil. Further, if only raw vegetable oil is to be used, it needs to be purified to reduce viscosity and impurities only then can it be used to run the engine.

Based on his studies unpurified biodiesel (coconut) could not run the engine. Purified (washed) biodiesel (coconut) ran a bit better than when compared to diesel, the emitted smoke was less and the engine was running smoothly. However, blending maybe more cost effective – diesel with purified biodiesel.

Discussion

- Engine durability has to be checked at the Gurugoda site. (Due to the low supply of oil to the laboratory at the Faculty of Engineering, University of Ruhuna, only about 20 minutes of engine running was done. About 300 millilitres of purified 100% biodiesel oil is required to run the engine producing 1kW for one hour.)
- Mixing percentage – what is the maximum (engine manufacturers say upto a 20% mix) point at which the engine output will go down if the biofuel mix is too high?
- In the near future research should be done using the existing engines – adjusting/modifying engines so that they can be used for biofuel running (looking into aspects such as the injectors, fuel pumps, etc). In such a case it maybe necessary to concentrate on certain types of transport modes (for example tractors, boats, etc).
- Can the oil specifications be changed/adjusted? Is it cheaper than modifying the engine? More investigation needs to be conducted to find injectors and fuel pumps suitable for using SVO in diesel engines. SVO can then be used in diesel engines and the cost of production will be very low as the esterification process will not be required.
- Given the costs of biodiesel (not oil expelling) and the inefficiencies is it feasible? Further studies should be conducted to find economical methods of producing biofuel in order to compete with diesel oil. It is necessary to go to the next stage of testing at the community level and for simple applications. Other simpler uses of biofuel – replacing kerosene lamps and cookers, candles, etc need to also be considered.

6

CHAPTER 6

FUTURE DIRECTIONS OF THE LIQUID BIOFUEL PROJECT

Liquid biofuels come in the form of biodiesel and ethanol through processed plant oil and starch respectively. In Sri Lanka the oil seed potential is yet to be identified however some plants present in Sri Lanka have been identified globally as viable liquid biofuel sources. Practical Action has adopted a self imposed policy not to touch edible crops for producing biofuels, and to promote only non-edible crops for the purpose. The initial stage of Practical Action's Liquid Biofuels project is to investigate a range of biofuel production potentials and applications as a renewable energy option for the poor in remote off-grid areas of Sri Lanka and elsewhere. To this end the project has conducted an audit on the current liquid biofuel status. Studies have also been carried out in the areas of energy plantation, extraction and processing of biofuels and application of such technology. It is these studies which have been compiled into this publication. The next stage of the project is the practical implementation of these processes at a community level as a pilot demonstration.

The project focuses on the studying of biofuel as an energy option for rural application as well as an option for the present oil crisis. Burning of fossil fuels harms the environment in the long run with greenhouse gas emissions, contributing towards other forms of environmental pollution and resulting in climate change.

Demonstrations of community based technologies and processes in biofuel generation, subsequent lessons and technology adaptations made are all project activities which are being undertaken in collaboration with researchers in the country. As a result of these initiatives there is scope to improve the technology further and increase its replication in other rural areas of the country.

6.1 FUTURE GOALS

The future goals of the project's demonstration interventions are to influence communities to have their own modes of energy provision. The other implementing stakeholders involved with the project will include the project aspects into their agendas so that the project objectives would be propagated through the stakeholders' interventions, independent of the project. The project will share the findings with a wider audience of implementers, academics and other interested audiences.

Manufacturing of equipment for the energy units as well as unit operators will obtain direct employment. 'Fuel' suppliers for community based

units can gain direct employment. Opportunities for income generation activities within benefiting communities will increase with increased access to power as does the potential for better education facilitated due to lighting and the powering of communication equipment (TV, radios, etc.) within rural areas.

Three universities (Peradeniya, Ruhuna and Moratuwa) in Sri Lanka, the National Engineering Research & Development Centre (NERDC) and Sangrama (partner organisation) and other members of the TAB joined hands with Practical Action's liquid biofuels work on the project from inception. They are contributing in a variety of areas such as crop science, biofuel plantation, oil expelling, fuel processing, applications of biofuels and facilitating community participation. The TAB on Liquid Biofuels, which is an external advisory committee established by Practical Action is also guiding the project interventions with their different expertise with national perspectives.

The project is expected in the long run to benefit those who lack access to electricity – providing them with a clean energy source to get electricity as well as thermal applications. Liquid biofuel power schemes involving the harvesting of energy plantations have direct income benefits for communities. Within the project period of 5 years (commencing 2007), about 200 families are expected to obtain electricity and water supply from such schemes. In addition it is expected to use biofuel as a transport fuel to benefit 250 families.

The liquid biofuel project is expected to have direct impacts on about 06 research institutions and 100 families in the forms of maintaining energy plantations (such as jatropha, other oil seed plants). These in turn are expected to have impacts on over 100 engineering, sociology, forestry, agriculture and botany students. Those who are trained and their capacities built, enhancing their skills and competencies, will have the potential to gain a competitive advantage in employment and obtain enhanced levels of incomes, particularly for those who would directly engaged in liquid biofuels related service provision.

In addition, schools (children and teachers) in the project area have been made aware of energy related issues, renewable energy solution focusing on biofuel applications.

The project will be implemented in Orissa, India (to learn and share experiences and lessons) and implemented in Rasnayakapura Divisional Secretariat, Sri Lanka. Subsequent to the studies conducted and included in this publication the project has moved on to the next stage of implementation where a community in Gurugoda has commenced planting and harvesting bio-energy crops.

6.2 FUTURE ACTIVITIES

The main activities planned for the future are,

- Studies on market chains, status of food security, energy plantation (types of crops, farms and lands), processing and applications (mixing with other fuels and allied engine performances), competition for land use for food crops, comparative costs of energy sources (Economic and financial costs and models)
- Community based biofuel plantation (as live fence), oil expelling, fuel processing and application (trial) demonstration in Nikaweratiya
- Initiate a dialogue with policy makers and practitioners on impacts of biofuels (including effects on climate change, employment opportunities, and business viabilities etc)
- Follow up the existing plantation studies and widen the operations for the areas such as mixed cropping.
- Improve the oil extraction and expelling techniques to get more oil yield while reducing the energy input.
- Carry out more research work to improve the biodiesel processing mainly targeting to reduce the cost of biodiesel production.
- Several new applications would be tested during the project period such as
 - i. Performance testing on diesel and biodiesel blending in existing engines without any major modification
 - ii. 100% biodiesel applications with existing engines
 - iii. Study the engine modifications for biodiesel and raw oil applications
 - iv. Introduce biofuel / raw oil direct applications such as for lighting lamps, cook stoves and some by products.
- Policy level dissemination and advocacy of results to ensure sustainability and movement towards community based renewable energy systems
- Cross learning across the region and dissemination of results of the project through networks, across the Practical Action group, as well as at international level.
- To assess the feasibility of community owned, operated and governed alternate green forms of energy and transport solutions leading to reduced emission of greenhouse gases
- To set up 2 sustainable community based energy producing and use schemes demonstrating and educating the public the effectiveness of community based energy and transport solutions.

ANNEXURE 1

METHODOLOGY FOR BIODIESEL PROCESSING FROM RAW VEGETABLE OILS

Oil Purification

Raw oils contaminated with suspended particles, moisture and FFA could interfere with chemical reactions and subsequent purification stages. Therefore, these ingredients are recommended to be removed before the transesterification of oil.

Suspended matters in oil can be removed by gravity settling or filtration.

Water is removed because its presence causes the formation of triglycerides. Splitting of oils and fats by hydrolysis, yields fatty acids; with glycerin as a by-product. Fatty acids under basic conditions promote saponification which will interfere in the process of glycerin separation. The problem can be rectified by heating the filtered oil to approximately 120 °C to boil-off dissolved or suspended water. When the water boils, it spatters. To prevent injury, this operation should be done in a sufficiently large container (at most, two thirds full) which is closed but not sealed.

Another method to moisture removal is to stir the oil with a drying agent such as magnesium sulfate to remove the water in the form of water of crystallization.

The drying agent can be separated by decanting or by filtration. However, the viscosity of the oil may not allow the drying agent to mix thoroughly.

Two methods are available for FFA removal. Soap formation and filtration is one of the methods. In the second method FFA are converted to the respective ester by reacting with an alcohol in the presence of an acid catalyst (refer reaction given in Figure 4.3, Chapter 4).

Acid Value

After extensive research carried out in the CPE laboratory at University of Peradeniya, it was decided that the acid value is the main parameter that will help to determine a suitable method for biodiesel processing.

Standard Test Method for Acid Number by Color-Indicator Titration is given by ASTM D974-06. However, due to lack of laboratory facilities available at the proposed biodiesel processing centre and also the accuracy is not a critical consideration, a simple method applied by US home biodiesel brewers was adopted. Nevertheless, this method is similar to ASTM D3339-04 (Standard Test Method for Acid Number of Petroleum Products by Semi-Micro Color

Indicator Titration). To determine the acid number, the oil sample is dissolved in isopropyl alcohol and is titrated at room temperature with standard alcoholic base solution to the end point indicated by the color change of the added phenolphthalein solution.

Apparatus

1L volumetric flask, watch glasses (02 nos.), electronic balance, spatula, CH₃OH containing wash bottle, 25 ml burette, glass funnel, 100 ml volumetric flask, 100 ml measuring cylinder, 100 ml reagent bottle with glass lid, 500 ml reagent bottle with plastic lid

Reagents

Reagent grade chemicals shall be used in all tests.



Ethanol with 95% purity



deionized water

IPA

Analytical grade isopropyl alcohol

1% NaOH solution

Dissolve 1g of NaOH in 1L of distilled water and shake till solid disappears.

Phenolphthalein indicator solution

Dissolve 0.5g phenolphthalein in 85 mL of 95% ethanol and dilute to 100 mL with deionized water.

Procedure

1. Fill 0.1% NaOH solution into a vertically fitted 25 ml burette

2. Measure exactly 10 ml of IPA in to a 50 mL beaker
3. Add two drops of phenolphthalein solution and mix well
4. Titrate with 0.1% NaOH solution until colour changes from colourless to pink (magenta). Report the burette reading (B)
5. Measure exactly 1 ml of oil and add to solution in step 4, and mix until it resulted in a single phase solution
6. Titrate with 0.1% NaOH solution until the colour changes from colourless to pink (magenta). Report the burette reading (A)

$$\text{Acid number} = \frac{A - B}{V\rho} \%$$

where

A = volume of NaOH used to reach end point (ml)

B = volume corresponding to IPA titration (ml)

V = volume of sample (ml)

ρ = density of the oil (g/ml)

Oil Processing

There are two processing methods have been tested; one-step and two step methods.

One-Step Method

If the acid value determined by method given above is less than 3.5 ml NaOH/ml, then the one step method may be utilized.

Reagents

Commercial grade chemicals (GP) shall be used.

CH₃OH

General Purpose methanol containing less than 0.5% moisture

NaOH

General Purpose sodium Hydroxide

Apparatus

Main Reactor, CH₃ONa reactor, watch glass, electronic balance, spatula, glass funnel, 200 ml measuring cylinder, Settling Tank, Crude glycerin storage tank, Crude biodiesel storage tank

Procedure

A general procedure for processing 1L of oil is as follows:

- 1) Accurately measure 1L of the oil into the reactor
- 2) Heat the oil to 55 °C
- 3) Dissolve 3.5 g of NaOH in 200 ml of CH₃OH to prepare CH₃ONa
- 4) Add CH₃ONa into oil and stir for 1 hour 600 rpm. Keep the temperature at a constant level throughout
- 5) Transfer the product mixture into a settling tank and allow settling for 24 hours
- 6) Carefully drained-off the bottom layer (mainly consisted with glycerine and impurities)
- 7) Store the top layer as crude biodiesel

Two-Step Method

Difficulties were experienced with alkaline-esterification (one-step method) of oils having large amounts of FFA. It was reported that the FFA quickly react with the alkaline catalyst to produce

soaps that inhibit the separation of the ester and glycerine. A two-step transesterification process is developed to convert the high FFA oils to its mono-esters. In the first step, acid catalysed esterification reduces the FFA content of the oil to less than a reasonable amount. The second step is alkaline catalysed transesterification process which converts the products of the first step to its mono-esters and glycerine.

If the acid value determined by method given above is more than 3.5, then the two-step method is recommended.

Reagents

Commercial grade (GP) chemicals shall be used.

CH₃OH

General Purpose methanol containing less than 0.5% moisture

H₂SO₄

Conc. Sulphuric (98%) acid

NaOH

General Purpose Sodium Hydroxide

Apparatus

Main Reactor, Esterification Reactor, CH₃ONa reactor, 25 ml pipette, Pipette Filler, watch Glass, electronic balance, spatula, glass funnel, 200 ml measuring cylinder, Settling Tank, Intermediate Product Settling Tank, Crude Glycerin Storage Tank, Crude biodiesel storage tank

Procedure

A general procedure for processing 1L of oil is given below:

- 1) Accurately measure 1L of the oil into the reactor
- 2) Heat the oil to 55 °C
- 3) Prepare 1% (by volume) H₂SO₄ acid in 250 ml CH₃OH solution
- 4) Mix the solution in step 3 with heated oil and stir at 600 rpm for 1 hour. Keep the temperature at a constant level (55 °C) throughout
- 5) Transfer the product to the Intermediate Settling Tank and allow 24 hours for the mixture to separate
- 6) Separate the impurity layer and put the esterified oil back into the Reactor after quantifying the acid value (A)
- 7) Dissolve (3.5+A) g of NaOH in 200 ml of CH₃OH to prepare CH₃ONa solution
- 8) Add CH₃ONa solution into oil and stir for 30 minutes at 600 rpm. Keep the temperature at a constant level (55 °C) throughout
- 9) Transfer the product mixture into a settling tank and allow settling for 24 hours
- 10) Carefully drained-off the bottom layer (mainly consisted with glycerine and impurities)
- 11) Store the top layer as crude biodiesel

Methanol Test

The biodiesel should be fully soluble in methanol, forming a clear bright phase. If not, there is pollution left (di- or tri- glycerides) in the biodiesel.

Reagents

Water-free methanol

Apparatus

250 ml separation funnel, 400 ml beaker, magnetic stirrer, electronic balance accurate to 0.05 g, 50 ml E-flask with narrowed neck

Procedure

- 1) Measure exactly 225 g of methanol into a beaker.
- 2) Add exactly 25 g of the biodiesel to the same beaker.
- 3) Stir the fluids for 2 minutes using magnetic stirrer.
- 4) Take the beaker off the stirrer and pour the contents into the separation funnel.
- 5) Measure the mass of any oil phase separate out from the biodiesel/methanol phase
- 6) Calculate the mass% of unreacted oil result as

$$\frac{m_1}{m_2} \times 100$$

where m_1 is the undissolved material and m_2 is the initial sample weight.

Wash Test

This is the most useful all-round test. The result will indicate whether the reaction is completed and a quality fuel has been produced without much impurities in it.

Reagents

Deionised water – 150 ml

Apparatus

500 ml measuring cylinder, 500 ml reagent bottle

Procedure

- 1) Pour 150 ml of unrefined biodiesel in a 500 ml reagent bottle
- 2) Add 150 ml of water, close the lid tightly and shake up and down violently for 10 seconds or more
- 3) Let the mixture settle for half an hour

If the biodiesel separated from the water with amber biodiesel on top and milky water below, the fuel is of high quality with minimal contaminants. If so, the crude biodiesel passes the wash test, and therefore can proceed with washing for purification. If it does not separate, or separates very slowly with a creamy white layer sandwiched between water and biodiesel the process needs improvements. Reasons for the observation may be due to excessive catalyst usage, or a poor conversion (less CH_3OH , less mixing or lower temperature than required) has lead to half-processed mono- and di-glycerides, fuel contaminants that act as emulsifiers.

Biodiesel washing

Biodiesel may be required to wash before use to remove soaps, excess CH_3OH , residual NaOH , free glycerin and other contaminants. However, some reports argue that washing is

not necessary as small amounts of contaminants do not cause engine damages.

Various washing methods were proposed in literature. Bubble washing was tested at the CPE laboratory. Bubble-washing was developed at the University of Idaho and is popular among small scale biodiesel brewers.

Reagents

Deionised water

Apparatus

Aquarium aerator pump with a ceramic bubble-stone, Washing/Settling tanks

Procedure

- 1) Pour a measured volume of glycerin free biodiesel into the washing/settling tank.
- 2) Add water (1/3rd of biodiesel volume) to the wash tank
- 3) Bubble air for ½ an hour from the bottom of the tank using aquarium aerator pump and bubble stone
- 4) Allow the mixture to settle for a sufficient time.
- 5) Remove the water layer via the bottom-drain.
- 6) Repeat steps 2 to 5 for 2 more times

Washing is completed when the water is clear after settling, with a pH of 7.

ANNEXURE 2

BIODIESEL PROCESSING FROM RUBBER SEED OIL (RSO)

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Introduction

The production of biodiesel from refined edible oil is popular because of low cost of chemicals and easy processing. However, due to escalation of food prices which mainly attributed to biofuel production from edible sources, the use of non edible oils are being investigated as feed stocks. These oils are generally reported with high free fatty acid (FFA) content, making the conversion process more difficult, time consuming and expensive. Non edible oil sources such as, castor bean, jatropha, neem and rubber seed are freely available in Sri Lanka.

Total rubber cultivated lands in Sri Lanka is about 116,478 ha. The annual rubber seed production potential is about 150-400 kg/ha from which only negligible quantity is utilized for re-plantation (Ramadhas et al., 2005; Dong, 2003). The oil content of rubber seed kernel is reported to be as high as 45%, but the oil yield from rubber seed with the hard shell is only about 20%. If RSO is converted into biodiesel with a conversion efficiency of 80%, annually 25-65 kg of biodiesel can be produced from a hectare of rubber plantation. This study focuses on

optimization of chemical usage in biodiesel processing from rubber seed oil.

Materials and method

RSO was supplied by the NERD centre. All the other chemicals (H_2SO_4 , Methanol, Iso Propile Alcohol, and NaOH) used were of commercial grade, purchased from the local market.

Initially RSO was transesterified directly with the use of an alkaline catalyst, but was unsuccessful. Therefore, it was processed in two steps: acid catalyzed esterification followed by based catalyzed methanolysis (Veljkovic et al., 2006). Experimental set ups were prepared using 500 ml reagent bottles as laboratory scale reactors. A constant temperature water bath was used to heat the reaction mixtures while mixing was done manually at regular intervals, consistently.

Acid-Catalyzed Esterification (1st Step)

98% Sulphuric acid was used as the catalyst in order to reduce the FFA content of RSO.

Predetermined quantities of methanol (in excess amounts) and H_2SO_4 acid mixtures were added to 200 ml of heated rubber seed

oil samples (60°C) and stirred for 1 hour. Different oil:methanol: acid ratios were investigated and results were given in Tables 1 and 2. After the reaction period, each mixture was poured into a separation funnel and the products were allowed to separate by gravity for at least for 12 hours. Quantity and FFA content of esterified oil fractions (intermediate product-IMP) were measured.

Base Catalyzed Transesterification (2nd Step)

Base catalyzed transesterification of the esterified oil fraction was the 2nd step of the process. Pre-estimated quantity of NaOH pellets (3.5 g/l of oil) were dissolved in methanol (200 ml/l of oil) and the mixture was added to heated IMP at 60°C. The mixture was stirred at a constant temperature for 30 minutes. Then the product was gravity separated. After the separation, crude glycerin layer was drained out and the FFA and yield of the crude biodiesel fraction was measured. The quality of biodiesel was assessed by wash test (Biodiesel, 2008).

Biodiesel Washing

Washing of biodiesel was performed with slightly acidic water by the method of bubble wash technique proposed by University of Idaho (Kalpage et al., 2008). Crude biodiesel was washed at least three times, using wash water 1/3 of oil volume. Five important properties of refined biodiesel was measured.

Results and discussion

Direct transesterification of RSO by methanol with an alkaline catalyst resulted in soap formation which was attributed to the high FFA content of oil (22.3%), therefore the two-step method was used. Esterification reaction (the 1st step) resulted in two layers; the first with impurities, excess reactants and reaction by-products and the second (the IMP- intermediate product) with mono-esters and more of try-acyl-glycerides. Table 1 gives the IMP volumes after esterification of 200 ml RSO with different methanol and H₂SO₄ volumes. Subscripts T and B denotes the position of the IMP layer as top or bottom in the separation funnel.

Table 1. Quantities of IMP

Methanol Amount/ ml	H ₂ SO ₄ /ml			
	1	2	3	4
20	150 _T	180 _T	194 _T	194 _T
30	165 _T	185 _T	195 _T	200 _T
40	170 _B	180 _T	160 _T	195 _T
50	175 _B	185 _B	190 _T	200 _T
60	210 _B	190 _B	200 _T	200 _T
70	205 _B	195 _B	190 _B	200 _B

Note: T- Yield settled to top
B- Yield settled to bottom

Separation of impurity layer to bottom was assumed due to the increased density of the fraction due to its composition. When this layer contained with lesser amount of excess methanol (a light weight component) and of water that formed during the reaction, the net effect of the

phase on density caused it to settle to the bottom. The reverse was observed when higher amount of excess methanol was used.

FFA values resulted after acid esterification are given in Table 2. It can be seen that the FFA value decreased as the reactant volumes (both methanol and acid) were increased.

Table 2. FFA of IMP

Methanol /ml	H ₂ SO ₄ /ml			
	1	2	3	4
20	7.4	6.3	4.9	4.7
30	6.3	3.4	1.9	1.5
40	4.7	4.7	1.8	1.8
50	1.9	1.4	0.9	0.7
60	1.9	1.3	0.9	1.3
70	2.1	1.2	0.9	0.9

When the methanol amount is too low, sufficient amounts of FFA were not converted into its monoesters, therefore high FFAs were resulted. IMP samples with highest FFA contents (hatched cells in Table 2) were completely solidified in subsequent alkaline transesterification due to soap formation. Transesterification of esterified oil with FFA values between 1.5-5% showed the expected separation after the reaction, but in some of the instances the two separated layers were solidified when kept in contact for prolonged period. Although those non solidified biodiesel samples (raw biodiesel) recorded an acid value less than 0.3%, they were failed in the wash test. Samples with FFA value reduced to below 1.5% in the

esterification reaction were able to successfully convert into clean biodiesel after washing with slightly acidified water.

After visual inspection and few preliminary tests such as, wash test and methanol test (Biodiesel, 2008) of the refined rubber seed biodiesel, the volume ratio of oil:methanol:acid for a guaranteed fuel was selected as 100:25:1.5. Shown in Table 3 is a comparison of four important properties of raw RSO, raw biodiesel and refined biodiesel with the American standard for biodiesel; ASTM D6751.

Table 3. Comparison with standard values

Property	Biodiesel			ASTM-D6751 Standard
	Raw oil	Raw	Refined	
Volume (ml)	1000	950	845	
Acid value (mg KOH/g)	44.6	0.2	0.3	<0.5
Density (g/cm ³)	0.943	0.906	0.906	0.88-0.94
Moisture Content (w/w %)	0.08	0.03	0.032	< 0.05
Viscosity at 40°C (mm ² /s)	66.2	8.06	5.82	1.9 - 6

Conclusion

Conversion of RSO into its biodiesel was studied. FFA content of the principal raw material (oil) determines its suitability as a biodiesel feedstock. Since the RSO recorded a high FFA content

(22.3%), the two steps method; acid-catalyzed esterification followed by base-catalyzed transesterification was applied.

Increased use of methanol and acid dosages in the esterification reaction (1st step) reduced the FFA of intermediate product (IMP), but this can be converted successfully into biodiesel only when the FFA of the IMP is below 1.5. Considering the product quality and cost, a formula for guaranteed RSO based biodiesel was presented as follows:

1st Step (Esterification):

Oil: methanol: H₂SO₄ = 100 ml: 25 ml:
1.5 ml

2nd Step (Transesterification):

Oil: methanol: NaOH = 100 ml: 20 ml:
0.35 g

The above formula converted 85% of rubber seed oil into its biodiesel.

Four important properties of refined biodiesel were compared with ASTM D6751 standard.

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ANNEXURE 3

INFLUENCE OF OPERATING PARAMETERS ON THE PRE-TREATMENT OF *Azadirachta Indica* (NEEM) SEED OIL AS A FEED STOCK FOR BIODIESEL PRODUCTION

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Introduction

Azadirachta Indica (Neem) is an ever green tree which is endemic to Indian subcontinent and has been introduced to many other areas in the tropics. Neem is valued as a natural insect repellent and pesticide. The oil of neem seed is used for skin care and skin disorders.

Biodiesel is a clean burning alternative fuel, produced from renewable resources of vegetable oils and animal fats. Biodiesel offers many environmental, economic and social benefits. Currently neem oil is experimented as a feed stock for biodiesel production (Venkateswara et al., 2008). Neem oil is extracted from seed by mechanical pressing or solvent extraction. The neem seed oil comprises mainly of triglycerides and triterpenoid compounds. The fatty acid composition of neem oil has a vast variation: main chemical components are palmitic (16 to 34%), stearic (6 to 24%), oleic (25 to 58%), and linoleic (6 to 17%) acids (Kaushik et al, 2000).

Neem oil has Free Fatty Acids (FFA) above 13%; the value varies with many factors such as the origin, age, and expelling method. Acid esterification is required prior to alkaline esterification for oils with high FFA content (>2%) (Gerpen et al., 2004). This research is focused on the reduction of FFA content of neem oil as a pretreatment step to lower the value below 2%.

Materials and methods

Materials: Neem seed oil was supplied by the National Engineering Research and Development (NERD) Center of Sri Lanka. All the other chemicals used were of commercial grade, purchased from the local market.

Method: Neem seed oil samples were reacted with predetermined volumes of methanol in the presence of concentrated H₂SO₄ as an acid catalyst at controlled temperatures. Reactions were carried out in 500 ml reactors (Pyrex reagent bottles) using an electric heater-magnetic stirrer arrangements (IKEDA Scientific Co. Ltd). The main objective was

to study the effect of different process variables (methanol and acid quantities, temperature and reaction time) on the reduction of FFA content during acid esterification. Mixing rate was kept constant for all the reactions.

Acid value of oil was measured by titrimetric method using 1% NaOH solution (Biodiesel, 2008) and the FFA content was estimated in terms of free oleic acids;

$$(\text{FFA} = 0.7883 \times \text{AV}\%) \quad (1)$$

Methanol and acid quantities for the esterification reaction were initially estimated from the equations suggested by Gerpen et al. 2004 as 40 ml and 0.5 ml, respectively.

Effect of Temperature: In each of the 5 reactors 200 ml of oil was preheated to predetermine temperatures (30, 40, 50, 60, and 70°C) prior to the addition of 0.5 ml of concentrated H₂SO₄ in 40 ml methanol solution. Mixtures were continuously heated to maintain the temperatures at the desired levels given above. Small samples (about 3 ml) were withdrawn from the reactors and FFA contents were determined at different time intervals.

Effect of Methanol Content: Methanol contents were varied (30, 40, 45, 50, and 60 ml) while keeping the oil (200 ml), H₂SO₄ (0.5 ml), temperature (60°C) and stirring speed (counter 6) at constant levels.

Effect of H₂SO₄ Acid Content: H₂SO₄ acid contents were varied (0.2, 0.5, 1.0, and 2.0 ml) while keeping the oil, methanol, temperature and stirring speed at constant levels of 200 ml, 40 ml, 60°C and counter 6, respectively.

FFA Content of Esterified Oil after Different Reaction Intervals: Esterification reactions were conducted at Oil, Methanol, H₂SO₄ acid, temperature, and stirring rates at 200 ml, 40 ml, 0.5 ml, 60°C and counter 6, respectively. Duration of reactions varied (15, 30, 45, 60, and 120 minutes). Each product was allowed to separate under gravity for 12 hours. The FFA content of esterified oil fraction was measured.

Results and discussion

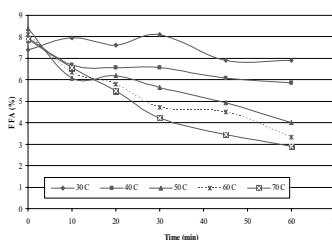


Figure 1. Effect of reaction temperature (Oil:Methanol:H₂SO₄ = 200:40:0.5 ml)

The FFA value of the neem oil used was about 14%. Figure 1 shows the result of temperature optimization study. It was observed that reaction rate considerably increase as the temperature increases. However, handling of lab-scale glass reactor became difficult above the boiling point of methanol (64.7°C).

Therefore 60°C was selected as the optimum temperature for the esterification.

Figures 2 and 3 present the effects of reactant volumes on FFA. The stoichiometric methanol requirement for the tested oil samples were about 25 ml. It was observed that the reactions progressed faster, when the excess reactant volumes increased. However, increased chemical usage would adversely effect on the cost of the product.

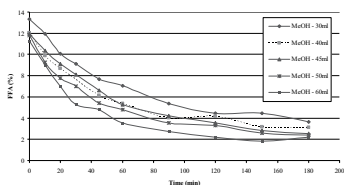


Figure 2. Effect of methanol content (Oil:H₂SO₄ = 200:0.5 ml, Temp=60°C)

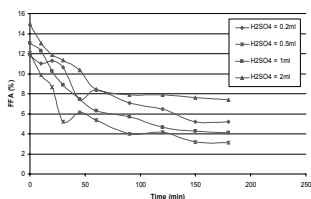


Figure 3. Effect of concentrated H₂SO₄ content (Oil: H₂SO₄ = 200:0.5 ml, Temp = 60°C)

It is important to note that the FFA values at t=0 in Figures 1, 2 and 3 do not coincide due to reasons of different dilution levels by reactants and non homogeneous nature of the oil. The resulting mixtures of the experiments shown in Figures 1, 2 and 3 were settled for 12 hours and product layers were separated

from impurities. The FFA level of the products are given in Table 1.

Table 1: FFA values of the separated products corresponding to the Figures 1, 2 and 3 after 12 hours of settling period.

Figure 1	Figure 2	Figure 3			
Temp variation, °C	FFA, %	Methanol variation, ml	FFA, %	Conc. H ₂ SO ₄ variation, ml	FFA, %
30	2.1	30	2.8	0.2 ml	2.9
40	1.9	40 ml	1.6	0.5 ml	1.6
50	1.6	45 ml	1.2	1 ml	0.8
60	1.5	50 ml	1.0	2 ml	0.7
70	1.3	60 ml	0.8		

Measurements in Figures 1, 2 and 3 were made while reactions were in progress. As a result, the FFA value in each figure replicated the combined effect of products which include triglyceride, esterified oil, unreacted FFA, excess methanol and acid catalyst. Therefore, FFA contents of esterified oil fractions were measured after 12 hours of settling. The reaction times were varied from 5 to 120 minutes. As can be seen in Figure 4, the FFA level of the product can be reduced below 2% in less than 40 minutes.

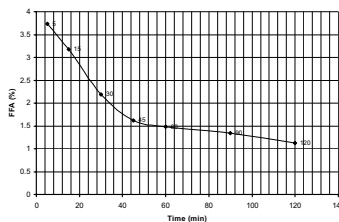


Figure 4. FFA content of separated product after different esterification periods (Oil:Methanol:H₂SO₄ = 200:40:0.5 ml)

Conclusions

Neem seed oil was studied as a feed stock for biodiesel processing. Since the oil has a high FFA content (>2%), acid esterification was performed as a pretreatment prior to alkaline transesterification. Rate of acid esterification was studied (in terms of reduction rate of FFA) at different operating conditions; reactant volumes (methanol and H₂SO₄) and reaction temperature.

Reaction kinetics improved with temperature; however 60°C was selected considering the operational difficulties in using higher temperatures. Although the increased reactant dosage (methanol and acid) increased the reaction rate, intern the chemical cost would also increase. It was estimated that if methanol volume was increased from 40 ml to 60 ml to pretreat 200 ml of oil, the cost would increase by Rs 25/= for the pretreatment of 1L of Neem oil (1L of Methanol = Rs 250/-). If concentrated H₂SO₄ volume was increased from 0.5 ml to 2 ml to pretreat 200 ml of oil, cost would increase by Rs 15/= for the pretreatment of 1L of Neem oil (1L of H₂SO₄ = Rs 2000/-). Esterification of 200ml of neem oil reaches the target reduction of 2% FFA content in less than 40 minutes. This Esterification was done with optimum mixture of 0.5 ml H₂SO₄ in 40 ml methanol at 60°C.

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ANNEXURE 4

BIODIESEL STANDARDS

BIODIESEL STANDARDS		EUROPE	GERMANY	USA	PETROLEUM DIESEL
SPECIFICATION		EN 14214:2003	DIN V 51606	ASTM D 6751-07b	EN 590:1999
APPLIES TO		FAME	FAME	FAAE	DIESEL
Density 15°C	g/cm ³	0.86-0.90	0.875-0.90		0.82-0.845
Viscosity 40°C	mm ² /s	3.5-5.0	3.5-5.0	1.9-6.0	2.0-4.5
Distillation	% @ °C			90%,360°C	85%,350°C 95%,360°C
Flashpoint (Fp)	°C	120 min	110 min	93 min	55 min
CFPP	°C	* country specific	summer 0 spr/ aut -10 winter -20		* country specific
Cloud point	°C			* report	
Sulphur	mg/kg	10 max	10 max	15 max	350 max
CCR 100%	%mass		0.05 max	0.05 max	
Carbon residue (10% dist. residue)	%mass	0.3 max	0.3 max		0.3 max
Sulphated ash	%mass	0.02 max	0.03 max	0.02 max	
Oxid ash	%mass				0.1 max
Water	mg/kg	500 max	300 max	500 max	200 max
Total contamination	mg/kg	24 max	20 max		24 max
Cu corrosion max	3h/50°C	1	1	3	1
Oxidation stability	hrs;110°C	6 hours min		3 hours min	N/A (25 g/m3)
Cetane number		51 min	49 min	47 min	51 min
Acid value	mgKOH / g	0.5 max	0.5 max	0.5 max	
Methanol	%mass	0.20 max	0.3 max	0.2 max or Fp <130°C	
Ester content	%mass	96.5 min			
Monoglyceride	%mass	0.8 max	0.8 max		
Diglyceride	%mass	0.2 max	0.4 max		
Triglyceride	%mass	0.2 max	0.4 max		
Free glycerol	%mass	0.02 max	0.02 max	0.02 max	
Total glycerol	%mass	0.25 max	0.25 max	0.24 max	
Iodine value		120 max	115 max		
Linolenic acid ME	%mass	12 max			
C(x:4) & greater unsaturated esters	%mass	1 max			
Phosphorus	mg/kg	10 max	10 max	10 max	
Alkalinity	mg/kg		5 max		
Gp I metals (Na,K)	mg/kg	5 max		5 max	
GpII metals (Ca,Mg)	mg/kg	5 max		5 max	

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