CHARACTERIZATION OF NON EDIBLE PLANT OILS AND THEIR USE FOR BIODIESEL PRODUCTION

By

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(2014- 2017)

Biomass energy.
Soxhlet apparatus.

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FINAL APPROVAL

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This humble effort is

Dedicated

To

My beloved

Parents

&

My late uncle

Abdul Mateen Qureshi

(May Allah grant him forgiveness)
Who inspired me for higher ideals of life

DECLARATION

I hereby solemnly declare that the work **"Characterization of non edible plants oils and their use for the production of biodiesel"** presented in the following thesis is my own effort, except where otherwise acknowledged and that the thesis is my own composition. No part of the thesis has been previously presented for any other degree.

Dated: 10-03-2017

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Muhammad Junaid

CONTENTS

ABBREVIATIONS

ASTM	American Standard for Testing Materials
A.V	Acid Value
cc	Cubic Centimeter
CNG	Compressed Natural Gas
EN	European Union Standard for Fuels
FAME	Fatty Acid Methyl Esters
FFA	Free Fatty Acid
I.V	Iodine Value
RPM	Revolution Per Minutes
S.V	Saponification Value

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ABSTRACT

Main issue of the society is energy due to its increasing demand and scarce supply. Today one of the major sources of energy is fossil fuel, but the fuel prices are increasing rapidly and burdening economy. These fuels are also harmful for the environment releasing greenhouse gasses causing climatic change. To resolve this issue renewable energy resource must be discovered and sustained to save environment and economy. In this study non edible plants like *Ricinus communis*, *Azadirachta indica* and *Pongamia pinnata* were used for the extraction, characterization of oil, transesterification and characterization of biodiesel. The oils from these plants were extracted by two methods mechanical and Soxhlet method. The extracted oil was analyzed for physical and chemical parameters. Soxhlet apparatus gave better yield than mechanical method for the selected plants. The extracted oil was transesterified via two step process using NaOH as catalyst at 70 °C for 1.5 hours. Biodiesel yield of transesterification process for *Ricinus communis*, *Azadirachta indica*, and *Pongamia pinnata* was 90%, 85% and 78% respectively. Specific gravity of oils and biodiesel was determined highest value for biodiesel was shown by *Ricinus communis* 930kg.m^{-3}. *Ricinus communis* oil had the highest viscosity 65cSt as well as its biodiesel 5.9cSt these value for biodiesel fall within acceptable limit of ASTM i.e 1.9-6. The acidic value of *Ricinus communis* oil was 10.4 mgKOH.g^{-1} and that of its biodiesel was 1.2 mgKOH.g^{-1}, *Pongamia pinnata* biodiesel showed acid value nearer to standard value for normal diesel which is 0.54mgKOH.g^{-1}. Iodine Value for all samples in case of oil and diesel didn't showed significant variation. Saponification value of *Azadirachta indica* oil was highest among the three samples i.e 198.5mgKOH.g^{-1}. Some of the physical and chemical characters shown by these plants fall under the specific range of ASTM limits.

INTRODUCTION

INTRODUCTION

Scientists have suggested that fossil fuels including natural gas, coal are main sources of power to perform routine work throughout the world but these fuels are non-renewable sources, once consumed cannot be reutilized and their emission is causing environmental hazards which is dangerous to human population. Their rapid consumption is due to the fact that the world population is increasing; also for the development of society energy is required. It is also necessary for transportation, domestic purposes, industrialization and growth of society. This situation has lead to the idea that world must seek resort from harmful aspects of limited energy resources and find renewable and eco-friendly fuels which are easily available (Barnwal and Sharma 2005). Non renewable energy resources like fossil fuels are limited in their availability, only few countries have abundant reservoirs of fuels. The cost of fossil fuels is increasing day by day; environmental pollution is also at maximum. These attributes of normal fuels are compelling the world to look towards biomass energy resources. It has all the beneficial features of an energy resource (Sensoz and Angın 2000). Bioenergy obtained from biomass as biofuels could be a promising solution for the future. Biofuels include biogas, bio ethanol and biodiesel. All these sources are renewable and have the ability to compete with the fossil fuel which is a dominant source of energy throughout the world. There is urgent need to bring in focus the importance of biomass with respect to biofuels (Barminas and Maina 2001).

It is a known fact that world oil production has declined considerably. The production of biofuels has increased 10 times due to high fuel prices and environmental impacts. There for the debate regarding the usefulness of biofuels and declining of fuel reserves has gained importance among political leaders and international organizations. But this fuel is not the complete solution as far as vehicle fuel is concerned. For global change requires the use of electricity in transport, solar energy, renewable energy resource and change of habits regarding fuel consumption. Much technological advancement is also required to take the biofuels production to the next level i.e production of biofuels from cellulosic material. More research and investment is required through biodiesel which indicate that energy yield of biodiesel is high. While energy yield of ethanol is 25% more than energy invested for its production. Biodiesel require less energy input for its production, releases less pollutant into the environment green house gas emissions are reduced to significant level of approximately 40%. At the moment any biofuel cannot replace petrodiesel even if all U.S soybean and corn production is dedicated for biofuels production it would only meet $1/10^{th}$ of gasoline demand and 6% of diesel demand. If the cost of petroleum increases it would ultimately increase the production cost of bio fuels. Bio diesel deserves the subsidy because of its environmental benefits. Bio mass grown on agriculture marginal land or waste land could prove to be promising source of bio fuel (Hill and Nelson 2006). Researchers have observed that there is an energy crisis in Pakistan, a large number of populations still have limited access to modern energy like electricity, and power shortage is frequent. This is due to the fact that economy of this country is not able enough to import a fossil fuel on a large scale. Known oil reserves of Pakistan are also scarce (Mirza and Maroto-Valer 2003).

In 2009 the Pakistani economy lost over 3.8 billion US dollars due to power shortages in industrial areas. Loss to GDP was about 2½, approximately 500,000 jobs were lost and export worth of 1.3 billion dollars was severely affected which hampered foreign exchange reserves of the country (Atadashi and Aroua, 2010). For the past few decades in Pakistan the consumption of energy has increased at very rapid rate. This increase is due to economical growth in the country. The supply of energy is not sufficient and is hampering economical development and industrial growth. The impact on environment is also negative. Thus, load shedding has become part of life in Pakistan. At this rate it would become difficult for government to meet the energy demand of people. This paves the way for exploring the non-conventional energy resources which are renewable. Reserves of natural gas are present in Pakistan but this gas is also being consumed at a very high rate. Pakistan uses its gas in vehicles as compressed natural gas which is available at many fuel stations. CNG is one of the major sources of energy in Pakistan domestically and industrially. But its reserves are depleting rapidly. Proper management of natural gas is required (Malik and Sukhera 2012).

Production of biodiesel using non edible oils can be used much more effectively than any other feed stock. Non edible oils are cheaper which can reduce the cost of production. Thus fuel produced is biodegradable and free of aromatic compounds and non toxic. This fuel has shown the capacity to compete with the fossil fuels and meet the challenges associated with normal diesel (Helwani and Otman, 2009). It is reported that in developed countries there is increasing trend towards using modern technologies and efficient bio-energy utilization using a variety of biofuels which are becoming cost wise competitive with fossil fuels. The biomass is converted via different methods into biofuels. Feedstock required is main determinant of compatibility with that of fossil fuels (Puhan and Vedaraman 2005). Research have found that plants such as palm, and other food related oil are used for the production of biodiesel but their productivity is low and vary large area is required for their plantation. Researchers now a day are using algae to produce oils which can be converted to biodiesel (Miao and Wu 2004). The world is searching for an environmental friendly fuel. To sustain the economy and society it is required to not only provide the world with sustainable carbon based fuel and to save the ecosystem from harmful effects of fossil fuels. Biodiesel is thought as one of the main alternative source of fuel. The situation is not ideal these methyl esters have high viscosity and cold flow properties are not up to the required standard for using in diesel engines. But its environmental aspects are beneficial emitting very low amount of green house gasses and chemically biodegradable (Srivastava and Prasad 2000).

Biodiesel can be produced from many types of vegetable oils waste cooking oil and animal fat. Its conversion into biodiesel involves alkali catalyzed transesterification reaction; sometimes this proves not feasible because free fatty acid content is high which leads to soap formation. For good yield and high value product it is first treated with strong acids (Van-Gerpen 2005). Biodiesel is in fact monoalkyl esters of fatty acid, produced by reaction of fat or oil, obtained either from animal or plant with an alcohol either methanol or ethanol in the presence of catalyst at a certain temperature. Glycerin as a byproduct is obtained which is a valuable product for pharmaceutical and food industry. They also reported that in this process excess amount of alcohol is required so that the reaction proceed in the forward direction for completion of reaction. At about 60 to 70 centigrade the reaction is completed in 90 minutes. A catalyst mostly alkali is also used in this reaction to enhance the rate of reaction and yield of product (Pinto and Guarieiro 2005).

Researchers know that diesel engine is one the most important mean of transport today. It can transport goods, people, generate electricity at domestic and industrial scale much more economically than any other machine in the world. But it is also the main cause of environmental problem all over the world, it will continue to be this way because the number of vehicles is rising with every passing day and the distances travelled by these vehicles is increasing. Its emission not only causes environmental problems but serious health issues as well like cardiovascular diseases, cancer and respiratory problems. Natural environment is being polluted which is causing the global warming, air, water, and soil pollution. To avoid such issues sulphur content and aromatic content is reduced which proves beneficial for environment. Also engine modification and development of new technologies is required but these developments take a long time to implement and control environmental issues (Lloyd and Cackette 2001). Biodiesel can be produced from vegetable oil or animal fat it can be used either in blend with petrodiesel or as pure diesel because it has very similar fuel characteristics and lower exhaust emissions. It is toxic compounds which can make the environment polluted. Some properties of biodiesel are much better than diesel which is renewability and biodegradability. Out of 350 identified crops only some of them are considered to be the potential feedstock capable of producing biodiesel at a large scale. There are some problems associated with plant oils one of them is high viscosity. There are normally four techniques applied like pyrolysis, micro-emulsification, dilution and transesterification to overcome the problem of high fuel viscosity. Problems associated with engine performances also exist. But when it is converted to biodiesel via transesterification their viscosity value decreases significantly. In fact all vegetable oils have high viscosity as compared to their corresponding methyl esters. Flash point of vegetable oils is also higher than methyl esters. After transesterification reaction the viscosity is lowered and density also seems to increase slightly. Either ethanol or methanol is used most frequently for the production of biodiesel. Methanol is used in this process due to its low cost. Methyl esters have much better environmental impact and are gaining popularity. These can be used in engines with minor or no modification (Demirbas 2005/a).

Alkali catalyzed transesterification reaction is much faster as compared to acid catalyzed and is most often used in commercial production of biodiesel. The process is influenced by temperature of reaction, presence of catalyst, water content of oil, reaction time and molar ratio of oil to alcohol. A molar ratio of 6 to 1 is required meaning 10ml of oil is dissolved in 60ml of alcohol for the reaction to proceed at optimum level (Ma and Hannah 1999). It is suggested by some scientist that the molecular structure of diesel molecule and fat molecule is nearly same. So it must contain same amount of chemical energy in its covalent bond between its two carbon atoms as biodiesel. As it is a scientific fact that energy is stored in the fat molecule of plant is of sunlight which is involved in photosynthesis. So the biodiesel produced will produce less harmful gasses than any normal diesel. This biodiesel produce less pollution. This is green fuel which degrades itself and does not persist in atmosphere or soil. Another factor which is to be noted that along with methyl esters it also produces glycerin which can be used for other purposes (Chisti, 2007). *Brasssica campestris*, *Helianthus annus*, *Glycine max* are some of the example of oils which are used as food and can also be converted to biodiesel due their availability. Third generation biodiesel can be produced utilizing algae. But one of the best sources is non edible oils, squandered oils from restaurants. This is because these are usually thrown away and not utilized for any fruitful purpose and is considered waste (Demirbas, 2008).

One of the non edible seed which produce sufficient amount of oil is Castor seed which is also known as *Ricinus communis*. This crop is economically beneficial due to high percentage of oil in its seeds. In a very short time this plant grows to its full length which is more than ten meters. This plant has the ability to produce seeds 360 days a year. The seeds are found in a capsule like body carrying seeds. The oil is golden color it is not fit for human use directly due to being purgatory in nature. Its oil contains mostly fatty acids which have one double bond in them while most abundant fatty acid is C18:1 ricinoleic acid (Chakrabarti and Ahmad, 2008). *Ricinus communis* is locally found in Africa and Subcontinent. The amount of investment required for the cultivation of this plant is very minimal but profit is many folds. In an area where there is very little rain or arid conditions it can grow easily. This plant has much more relevance than most of the plants of same traits. Its products are commercially profitable and are sold under different labels. Many techniques involving large scale production of some items utilize its products and oil. Another attribute is added to this plant due its utilization for the production of biodiesel. Hundred of uses are reported about this plant from herbal medicine to being involved in production of polymers. Due to its thermal properties it is considered as effective in many production units. Governments are trying to find a way to control oil prices and stabilize the environment and on the other hand want to earn profit as well. Large scale production of biodiesel using *Ricinus communis* is a great possibility. This oil has many uses besides fuel production which makes manufacturing of biodiesel a bit difficult. The properties shown by this oil shows that it is very much suited to biodiesel production (Salihu and Gana, 2014). It has been shown by researchers that this plant has very high herbal medicine value. Its products have been analyzed and profiled completely. It is believed that this plant can produce compounds which will cure many ailments in the future alongside many other benefits related to people (Jena and Gupta, 2012).

Another plant found abundantly in subcontinent and Africa is commonly known as Souk Chaeen (*Pongamia pinnata*). This plant grows very easily and attains maturity in a short period of time. From agricultural point of view this plant can be grown on land unfit for normal crop growth. This plant produces non edible oil not used for food due to high acidity value. The oil percentage of this crop is also very high which makes it suitable for biodiesel production. Land conditions required for the cultivation is not very specific and non edible oil is produced so its seeds can be used for the biodiesel production. The area covered by this plant is replenished and the soil is make more fertile and has good aesthetic impact on environment (Scott *et al.*, 2008). Fatty acid profile of *Pongamia Pinnata* shows that oleic acid is dominant in its oil. The oil percentage is also very high. It sheds about 200,000 seeds per decade each having an average weight of 1.7g. its seeds produce seed cake which is fed to animals. The oil is used as home remedies and as herbal medicine. Its wood is valuable and used for making many items like chairs and other house hold items (Gawali and Wagh, 2015). This plant has the ability to produce biodiesel at large scale. It can also produce such compounds which are biologically useful for human beings. This plant has many uses besides its beneficial aspect as bio energy crop. This plant has the potential to become main biodiesel feedstock in the time to come. This plant has very high percentage of oil which is why this can become a major fuel supply for the diesel engines (Parsada *et al.*, 2014).

Neem (*Azadircachta indica*) plant is native to Europe but now it has adapted successfully to Subcontinent environment and also East Asia. It reaches a height of about 30feet . (Kaushik and Vir 2000) showed in their study that Indian *Azadirachta indica* produces 700,000 tons of grains from which oil can be extracted. The percentage of oil in seeds is thirty percent. The most abundant acid found is oleic acid and over all it is approximately 70% unsaturated in nature (Awolu and Obafaye 2013). Neem plant is thought as one of the most valuable drug producing plant. This plant not only has high oil percentage it is used in many remedies like skin infection and small wounds. It is also used as pesticides. It has been shown that it is spermicidal in nature as well. Compounds prepared from this plant have been shown to treat heart related problems and malignant tumor (Sharma and Tomar, 2011). The idea that Neem oil can be used as diesel fuel is still in its early stages because its oil production is not much high. Hugh investment and investigation is needed to initiate the process which could sustain itself and produce energy. If processes related with production of biodiesel are improved and hurdles removed this plant has all the attributes to becoming energy producing crop (Ali and Mashud, 2013).

The oil from castor seed can be drawn out via different methods like solvent extraction and mechanical extraction. Dangerous chemicals are used in these techniques which can catch fire easily so precautionary measures are necessary for using solvent extraction method. This method is costly and dangerous so mechanical method is sometimes preferred (Hincapie and Mondragon, 2011).

The most abundant plant found in the local region is *Pongamia pinnata* it is found everywhere in parks gardens and in many houses especially in villages. This plant is high in oil percentage and can produce biomass required for the production of biodiesel. The plant named *Ricinus communis* commonly known as Arind in Punjab and Potohar region of Pakistan. This plant has much relevance due to its use in drug industries. Castor plant has also been identified as one of the plant which can provide biomass for biodiesel production. Similarly *Azadirachta indica* also fits the criteria described for the other two samples. Biodiesel production unit based on these oils can enhance economic activity in the region and will also result in prosperity of the region. In the present study the assumption was made by analyzing these plants and it became evident that these samples can be used for large scale production of biodiesel depending on the favourable traits discussed here.

1.1 Aims and Objectives

Following are the aims and objectives of this study

I. Production of oils using different techniques form the samples
II. Analysis of physical and chemical properties of oils
III. GC analysis of oils
IV. Biodiesel preparation and analysis of biodiesel

REVIEW OF LETRATURE

REVIEW OF LITERATURE

Agarwal and Das (2001) found that due to expansion of civilization and migration of people towards heavily populated areas of the world the usage of energy is at its peak. There is also improvement in automation of many processes related to large scale production which also utilize non-renewable energy. Increased reliance on only one source of energy has caused economical and environmental problem globally. Fossil fuels are limited and non-renewable; extreme use of it will result in an emergency situation regarding energy.

Demirbas (2002) showed that in alkali catalyzed transesterification reaction procedure, the alkali either KOH or NaOH is dissolved in alcohol which is either ethanol or methanol in a small reactor while shaking continues. The lipid is shifted is to a reactor and sodium metahoxide or sodium ethaoxide is added to it. The mixture is stirred vigorously at about 65 centigrade and time duration is about 120minutes under pressure. Two clear phases appear one is methyl esters and other is of glycerin in a successful reaction. These phases are separated via treatment with an acid to allow neutralization of alkali catalyst and then it is shifted to separating funnel and left for 24hours. Separation is complete when two phases appear, upper phase consist of methyl esters and lower of glycerin. The glycerin is collected from the bottom of the separating funnel. Biodiesel is washed to remove any catalyst and unreacted species like acid, alkali or glycerin. These materials are dissolved in the water and settle to the bottom which is then removed easily. The water is warmed to about 40 Celsius before washing of biodiesel. Afterwards it is dried to remove any moisture by heating at 373K.

Demirbas (2005/b) analyzed the production of biodiesel. He stated that the main function of the catalyst is to combine ethyl or methyl group to fatty acids to convert them into fatty acid ethyl and methyl esters. Glycerin obtained is an important by product. Transesterification process is used to reduce the viscosity of oil to enable its use in diesel engines. When methanol is used for the transesterification reaction to make methyl esters from straight chain fatty acids, then this process is called methanolysis. Most commonly catalyst used are potassium hydroxide (KOH) and sodium hydroxide (NaOH), these are homogenous catalyst the reaction goes forward under these catalyst easily. Some acids like HCl and H_2SO_4 are also used for the pre-treatment of oils and as a catalyst. Other heterogeneous catalysts like sodium methaoxide, sodium ethaoxide, sodium butaoxide are also utilized for maximum yield and optimum reaction performance. These heterogeneous catalysts are formed by making a solution of an alkali in its corresponding alcohol. Usually 1% sodium or potassium hydroxide solution is made in methanol, ethanol or butanol.

Attene and Falcidieno (2006) reported that from economical point of view fuels are required for not only construction of infrastructure also for the betterment of community as a whole. First world nations have abundant supply of fossil fuels due to strong economy or huge natural reservoirs. Third world nations face the problem of weak economy and insufficient reservoirs which creates the problem of fuel prices and presence in the market. To resolve this issue there must be a source which is not only beneficial for the atmosphere also inexhaustible. To live life in a modern way there must be plenty of fuel available all the time for the people around the world.

Bowman and Hilligoss (2006) in their study showed that many features of methyl esters are desirable like renewability, non toxic, availability, combustion efficiency, lower SO_2 and hydrocarbon (HC) content Biodiesel burns completely and thus gives clear flame because incomplete combustion gives rise to pollution. Because biodiesel has increased viscosity than normal diesel it can provide better lubrication which results in improving engine life.

Da-Silva and Maciel (2006) reported the definition of biodiesel. Biodiesel is defined by the American Society for Testing and Materials as mono alkyl esters of long chain fatty acids produced from lipids obtained either from plant or animal source.

Meher and Dharmagadda (2006) in their research work on *Pongamia pinnata* for biodiesel production and optimization of conditions for transesterification, concentration of catalyst and oil molar ratio. They found that methyl esters yield of this oil is about

96% on average. They calculated the acid vlaue of oil 5.06mg/g and whaen this oil was treated with KOH for conversion this acid value dropped to 0.6mg/g.

Ahmed and Lewis in (2007) suggested that the fear of polluted atmosphere and increasing fuel demand is thought as motivating force behind renewable resources. A fuel checking the bad impact of mineral oils must be present in the market to avoid dangers associated with normal diesel and its emissions. This concern of human beings must be alleviated through proper action.

Ahmed and Yokota in 2007 reported that fuels obtained from biomass are known as biofuels these are biogas, bioethanol and biodiesel. The idea that biofuels are a unique source of energy that can save economy and act as buffer to vanishing oil reserves is gaining popularity. There is also a great chance for the local farmers to grow energy rich crop and subdue the monopoly of fossil fuel in the market. Far off places where fuel supply is difficult can gain profit from this new source of income.

Akoh and Chang (2007) reported their findings about transesterification equilibrium reaction. They showed that the ratio of alcohol is kept higher to move the reaction to completion. This allow maximum ester yield. Some attempts were made to produce the biodiesel via immobilized lipases enzyme but the estimated cost for this type of production is not economically feasible.

Arndt and Simler (2007) showed in their work that the byproduct of biodiesel i.e. glycerin can be utilized by pharmaceutical and food industries in purified form. It can also be used for the manufacturing of polymers, lubricants and surfactants. In processes like fermentation it can be used as carbon feed stock Many sectors can gain benefits from this approach, first of all most benefit will be gained be agriculture sector due to increase of import and export thus leading to economic growth. It will create many jobs for unemployed people and by paving the way for new industries.

Chisti (2007) analyzed that surface production of micro algae is very high, lands not able to grow crops can be utilized to grow algae it does not compete for arable land which can be used for growing other crops. Because a micro-alga performs photosynthesis they produce environmental benefits and can result in reduction of green house gasses. Different types of species can be grown on different type of environments including fresh, sea water and waste water. Under stress conditions it can accumulate more than 50% oil per day weigh But during stress condition the accumulation of lipids inhibits cell division in algae which is a not desirable for microalgae producing biofuels.

Demirbas (2007) reported that biodiesel is produced via transesterification reaction. It is a reaction between an ester a triglyceride an alcohol in the presence of a catalyst to produce monoalkyl esters and glycerin. This process has proved to be a feasible source of biodiesel production. Biomass utilized for this purpose is renewable feedstock for this reaction. Chakrabarti and Ahmad (2008) analyzed that engines running on biodiesel produce less pollution than normal diesel and environmentally friendly. This attribute adds to the point that biodiesel production can prove to be feasible and productive.

Hribernik and Kegl (2007) reported that European Union has discussed about biofuels and has announced that after a decade or so each vehicle must be complied to use biofuels which would improve atmospheric conditions and the use of biofuels will be stimulated. The biodiesel can be used separately or mixed with other fuel. 80% of normal fuel will be mixed with biofuels chiefly biodiesel in all types of vehicle movements in Europe in next decades and a half. Canacki (2007) also stated in his work about biodiesel that it produces very less amount of green house gasses and dangerous compounds which pollute the atmosphere.

Demirbas (2008) again analyzed the importance and history of biodiesel. He showed it is a historical fact that first diesel engine designed by Rudolf Diesel was powered by vegetable oil. Viscosity of vegetable oil is very high as compared to normal diesel which is why it cannot be used directly in a diesel engine or even used as a replacement of fuel. To reduce the viscosity of oil transesterification also known as alcoholysis is performed, in this reaction oil and alcohol are reacted to produce esters and glycerine. It a reversible reaction excess amount of alcohol is utilized than that of oil. Catalyst like alkali is used to speed up the reaction and percentage of yield. In short transesterification is conversion of oil and fats to their corresponding esters.

Scott *et al.*, (2008) showed in their research that *Pongamia pinnata* is a leguminous plant which is nitrogen fixing and drought resistant. It has the ability to produce higher amount of oil and can be grown on lands not suitable for cultivation of normal crops. This plant can be a potential source of feed stock for the production of biodiesel. For large scale production of oil complete knowledge is required about genetics, physiology and environmental impact of the *Pongamia pinnata*. Focus of the research should be to maximize the production of oil from this plant as it is required for synthesis of biodiesel.

Cr-Deligiannis and Anastopoulos (2009) studied the impact of vegetable oils for the production of biofuels. They found that most commonly used edible oils for the production of biodiesel are rapeseed and sunflower which account for 97% of total production of biodiesel production. Soybean and palm oil accounts for only 1% each, this is due to their availability on large scale. But this practice is damaging the global economy due to the fact the demand of food is high. Also it raises an ethical question whether fuel is more important than fuel or not.

Demirbas in (2009) found that high octane and other mineral fuels are becoming costly and inadequate for use to boost the economy of any country. The dearth of these fuels is not in accordance with the people who think there must be plenty of supply of it so that there could be no loss of any kind. These mineral oils produce harmful gasses like NO_x ,SO_x and HC compounds when used in their respective engines. This severely pollutes the atmosphere. To resolve this matter a new fuel is to be discovered or prepared to stop environmental changes. In the future biodiesel can prove to be the solution of this problem.

Escobar and Lora (2009) Rural development can be reached if the development is sustainable which can be achieved through growing oleaginous crops. Life cycle analysis is used as a tool in this regard for growing such crops. It is concluded in this research paper that rise of oil prices in the future is a fact. Internationally establishing of rules and regulations, regarding the use of land for the production of biofuels is a necessity. Workers working to produce fuels must be given proper compensation and working environment.

Pinzi and Garcia (2009) conducted studies on non edible plant oil and found that there are a number of issues pertaining to biofuels still remain to be resolved like technical, social and economical issues. The manufacture of biodiesel has come under criticism and subsidies granted for its production have been condemned by many governments. The issue related to utilization of land for energetic crops like *Azadirachta indica, Terminalia catappa, Madhuca indica, Pongamia pinnata,* and *Jatropha curcas* must be sorted out. Biodiesel is alternative to normal diesel; the popularity of biodiesel must be promoted through social movements to allow governments to take the issue seriously and take necessary actions for the promotion and to remove hurdles in the way for renewable energy sources. Also, optimization of transesterification process must be done for each oil from which biodiesel can be produced; normally a general method is applied for most of plant oil which results in increase of cost of production. *Azadirachta Indica* and *Pongamia pinnata* oils fit European and US standards EN14214 and ASTM D675102. But both of them do not match all the properties of the fuel required for optimum engine performance. For ideal performance higher concentration of monounsaturated fatty acids like oleic and palmitic acid and lower concentrations of polyunsaturated fatty acid are recommended. For best performance in cold weather C18:1 and C16:1 acid are suitable. Genetic engineering can be applied to plants to produce oils that fit ideal fatty acid profile. For utilization of biodiesel in an acceptable way for all the components of societies a compromise must be reach between economical, social and technical factors.

Tilman and Socolow in (2009) found that for the production of biofuels it is necessary to find out which feedstock is best because it must not raise any issue regarding supply of food and environmental impact. To produce biofuels the governments must provide proper infrastructure, subsidies and monitoring of the production unit. Unauthorized production of biofuels can be disastrous for the economy and society in many ways. Training of the personals related to this field is also required to cope with improvements in technologies and production procedures. The chances for biofuels to become reliable resource are very high.

Ndana *et al.*, (2010) have conducted a research on *Jatropha curcus, Azadirachta indica, Ricinus communis,* Rubber for the production of biodiesel. These plants were analyzed in detail for the production of biodiesel. Transestrification reaction was carried out in two steps and both feedstoks edible oil and non edible oil were used in this study. They reported very high yield of close to 100% by using base of potassium. The fuel obtained was analyzed and it was found that this fuel is feasible for use in diesel engines. Petrodiesel standard values compared well with values found in this study.

Panwer and Shrirame (2010) reported in their research confirmed the feasibility of castor oil for use I diesel engines. They found that lower blends of castor biodiesel increase brake thermal efficiency and save fuel. Exhaust gas emission increased when the amount of biodiesel in blend was increased. The acid value for castor oil and that for biodiesel were determined and recorded as 1.642 mg/g for oil and for methy esters 1.08 mg/g.

Salimon and Noor (2010) studied Malaysian castor oil and its characteristics. They have shown that native castor is very high in oil content >40%. I_2 value and saponification valuesa are also very high. The acid vlue determined in this study was less than 5 mg/g and FFA% was around 4%. The HPLC was performed and it was found that this oil is about 98% saturated and fatty acid profile was analyzed. The ricinolic content was high in this oil so it was shown that this oil is quite suitable for biodiesel production.

Saribiyik and Ozcanli (2010) analyzed in their work that non edible characteristics of castor oil and high oil content make this plant suitable for biodiesel production. Cetane number of castor biodiesel is very high for this reason it can be mixed with blends having low Cetane number comprising of different biodiesel. Cold filter plugging point and pour point values of castor biodiesel was in agreement with EN 14214; addition of castor biodiesel lowers CFPP and pour point values because it has better cold flow properties. But it cannot be used in pure form in diesel engines because it has high viscosity and can be problematic for engines.

Bello and Makanju (2011) in their study conducted on castor oil showed that it is highly soluble in alcohol and require very low energy input and low catalyst concentration for biodiesel production which leads to economical production of biodiesel at industrial level. Its biodiesel burns completely because it's high oxygen content, resulting in 20% more load capacity than normal diesel.

Radha and Manikandan (2011) stated in their research that normal diesel produce green house gasses and nitrogenous compounds which are harmful for environment, whereas biodiesel produced by neem oil has the advantage that not only its use in engines is under accepted levels also it reduces emission of carbon monoxide and beneficial for environment. But reduction in nitrogen compounds is less significant.

Abdulkareem and Jimoh (2012) analyzed that there are many disadvantages related to mineral fuels one of it is climatic change and the other is exhaustibility. Due to green house effect the glaciers are melting and many forests and fertile lands have been destroyed. This fuel is also going towards its final stages these reservoirs will be dwindling in the coming future. It has also been find out by researchers that the rate of production is much less than the rate of exploit this fact confirms its short life span.

Aransiola and Betiku (2012) analyzed neem oil for use of tranesterification process which took place in two steps alkali as catalyst. They found that neem oil showed desnity 43.5cSt. and when this oil was converted into biodiesel about 7 times lower than for oil. These vlaues were compared with ASTM vlaues and it was found that they compare well with it. It was also shown by these resesachers that blends reduced the amount of green house gases when used in engines. Countries where this plant is in abundat can utilize this feed stock.

Bobade and Khyade (2012) also showed in their study that non edible plant like *Pongamia pinnata* is available in many parts of the worlds including subcontinent, it is much less costly than edible oils. There is a need to explore non edible oils for the production of biodiesel. By increasing the production of non edible oils like *Pongamia pinnata* oil the biodiesel can be synthesized in larger amounts. This can provide many economic, local, social, regional and environmental benefits. Some innovations are required to convert biodiesel into economically important alternative fuel. Pongamia oil contain FFA about 3% to 5%, rest is triglycerides and lipids. Viscosity of oil is another concern which is slightly high and may not flow properly from injector of engine. This is brought to an acceptable level by converting it into biodiesel. It is further reduced by blending it with normal diesel; also, flash point is higher than normal diesel which signifies its safety for use in domestic and industrial purpose. Incomplete transesterification and separation of biodiesel produce biodiesel of low quality. The reaction must be completed and separation must be carried out with care for high yield of biodiesel. For this reason the process needs to be optimized. FFA (free fatty acid) contents must be brought to a low level.

Ezeanyanaso and Ajibola (2012) conducted studies on storage of neem oil. They found that neem oil was not able to pass the biodiesel for storage purpose. Without additives most plant oils fails 6hr test. For storage up to a year additives are required but physicochemical properties change overtime also calorific value of biodiesel decrease due to storage with additive.

Khandelwal and Chauhan in (2012) stated about the debate related to the production of biodiesel is going on about its effectiveness as proper fuel. Since it is produced from food related crops and cannot be produced in large quantity at an economical price rendering the mineral fuel as dominant one. This debate has compelled scientist to find another source which is not food crop related. The attention of scientists has shifted towards nonedible oils which is second generation biofuels. This has advantage that these oils have no issue with food supply because acidity in these oils is high leaving them undesirable for use as food. Non edible oils are thought to have the ability to compete in the market with mineral fuel.

Okullo and Temu (2012) reported in their research that biodiesel from castor oil has very high viscosity therefore its use in diesel engines is not recommended. For reducing its viscosity it is blended with petro diesel. FFA content of castor is also high, which leads to lower yield of biodiesel and vice versa. For lowering FFA content improvements in neutralisation conditions is required because castor oil is sensitive to rigorous conditions such as high temperatures and high speed of mixing. It has been shown that neutralisation of castor oil is required for lowering FFA which improves not only the quality of biodiesel also increases its yield.

Venkatesan and Vikram (2012) analyzed the Pongmaia oil methyl esters and its blends. They reported that density of pongamia oil is 0.94g/cc and viscosity as 51.5cSt. the oil was transesterified by using oil and alcohol ratio of 6:1 a slight amount of sulphuric acid was also used in the first step. The reactionn time was 180min at 60 celcius. Other values such as Calorofic values and Cetane number was also measured in this sutdy which again was upto standard.

Abdulkadir and Adisa (2014) found that the physical and chemical parameters found in this study are slightly higher than those of standard values. Different types of cylinders were used to analyze exhaust emission while utilizing Neem biodiesel and normal diesel. One of the blends having 30% biodiesel has significantly lower exhaust heat loss and lower temperature. They found that the values of Neem biodiesel comparable to standard values. They also calculated acid value of oil at 6.77mg/g. It was also observed that hydrocarbon emission was also decreased when Neem biodiesel was used in the engines

AL-Harbawy and AL-Mallah (2014) carried out research on Castor biodiesel and its characterization. They extracted oil from castor via two different methods. One is hot method and other is cold method. They found that specific gravity 0.95, acidic value is 3.12, iodine value 74.08, Saponificaiton value 181.52, kinematic viscosity is 238 mm^2/s these values as considered around standard value for the hot method extracted oil. The oil produced by hot method was of much better quality. While some properties of cold method produced oil are according to standard value and some values are not according to standard.

Asmare and Gabbiye (2014) in their research about Ricinus communis and its characterization of properties such as physical and chemical. The oil was converted into methyl esters. The acid vlaue, iodine value, and saponificaiotn vlaue of oil were detrmined and compared with ASTM and EN14214. The esters yield form this oil was about 95%. The data obtain was analyzed statistically via software programme. The ester was high in density and viscosity which was brought down by blending it with diesel to meet the standards

Awolu and Obafaye (2013) also in their research conducted on neem oil showed that the physicochemical parameters which include moisture content, specific gravity, acid value, iodine value comply with the ASTM values which are necessary for proper engine functions. Physicochemical properties and its high yield of oil, verify that it could be a promising source of biodiesel production by using two step transesterification processes.

Mathiyazhagan and Elango (2013) found in their research that biodiesel produced from non edible oils like neem, needs to be tested first before authorization of its use in vehicles running on diesel. Different blends of biodiesel and petro diesel ranging from 10% to 50% were tested in single cylinder compression engine, two parameters break thermal efficiency (BTE) and specific fuel consumption of engines running on biodiesel blends and petrodiesel were studied and comparison was made. The results showed that both biodiesel blend and petrodiesel showed similar performances and that bio diesel produced by neem oil is viable for use in diesel engines.

Rayouf and Msaadaa in (2013) reported about edible oil and stated that the seeds of *Helianthus annuus*, *Brassica napus* and *Glycine max* are used in food. The oil from these sources can be converted to biodiesel. Plant oils which are used in food are not good for fuel production. The need for fuel does not advocate the utilization of food source for economical benefits. These fuels have their own importance but food crops are more suited for use as food.

Wu and Wang (2013) in their research showed that third generation biofuels are produced from algae mostly microalgae. It is a photosynthetic organism with high nutrition value, can be found living in salt water in sea and on land as well. Bio oil, biogas and biodiesel can be obtained from microalgae. It requires less cultivated land area and has short life time circle has made microalgae advantageous over other type of biofuels. It is being considered as an alternative to petrodiesel.

Bhuiya *et al.*, (2014) reported about the fact that soil which is suited for food crops and climatic conditions required for their optimum yield is not needed for non edible crops. Soil requirement are different for growing nonedible oil feedstock. To grow these crops minimal investment is required and profit is much more than the input which makes this project financially fruitful. These crops cover vast area replenish the environment makes the soil nutrient rich. The list of plants which fit the profile for the production of non edible oil is very long. This factor is meaningful when we try to know the importance of biodiesel produced form these crops. If the biomass which is oil from seeds of these plants is made less costly then there is a chance that biodiesel can be produced on a large scale. These traits of non food crops have focused attention of scientists and governments to produce biodiesel which has monetary benefits and does not hinder food supply rather supports it.

Doddabasava (2014) analyzed the production cost of pongamia oil. This scientist found that 60% of cost of production is due to feed stock seeds of pongamia and remaining is related to chemicals and other operation cost. After oil extraction two products are obtained, one is glycerin and other is seed cake. If both of these co-products are utilized properly the cost of production can be brought down by 21% by seed cake which can be used as animal fodder and 2% by glycerin.

Djibril and Lamine (2015) carried out work on converting oil of Neem into methyl esters using sodium hydroxide as catalyst under optimum circumstances. The fatty acid profile was also analyzed and four acids comprises most of the fatty acid present in the oil. The reaction kinetics was studied under optimized conditions. The reaction was carried out at 75 Celsius for 1.5 hrs to achieve maximum yield. Chemical and physical characterization was carried out by determining values such densities, viscosity, iodine value, Saponification value an acid value for both oil and methyl esters and compared with standard values.

Gokdogan and Eryilmaz (2015) worked on castor oil for biodiesel production using NaOH and alcohol. This oil is feasible for this purpose because its oil yield is high (approximately 50%) as compared to other oils. Different blends were also prepared and studied as well. It was found that as temperature increases the density and viscosity decreases. The blends of different types also show this trend prepared by mixing with petro diesel. In their study they found viscosity of castor oil as 212mm^2/s and that of biodiesel was 15mm^2/s.

Sriramajayam (2016) in his work on neem oil produced biodiesel using molar ratio of concentration of catalyst and oil as 1 to 7.5 12min. maximum yield of up to 98% was obtained. Viscosity of oil was measured as 5.68 mm^{2}s^{-1}. The acid value of 11.1mg/g, iodine value 75.5 gI_2/g and saponificaiton value was 203.4mg/g for oil were reported in this study. The characterization of Neem biodiesel was carried out by determining acid values, iodine values and Saponification values are 2.87mg/g, 77.2 and 198.5 respectively were reported.

MATERIAL &
METHODS

MATERIAL AND METHODS

This study was carried out at Pir Mehr Ali Shah University of Arid Agriculture Rawalpindi.

3.1 Material

3.1.1 Reagents and compounds

Every compound and reagent used was of analytical grade.

3.1.2 Plant species

Figure 3.1: Seeds of *Ricinus communis*

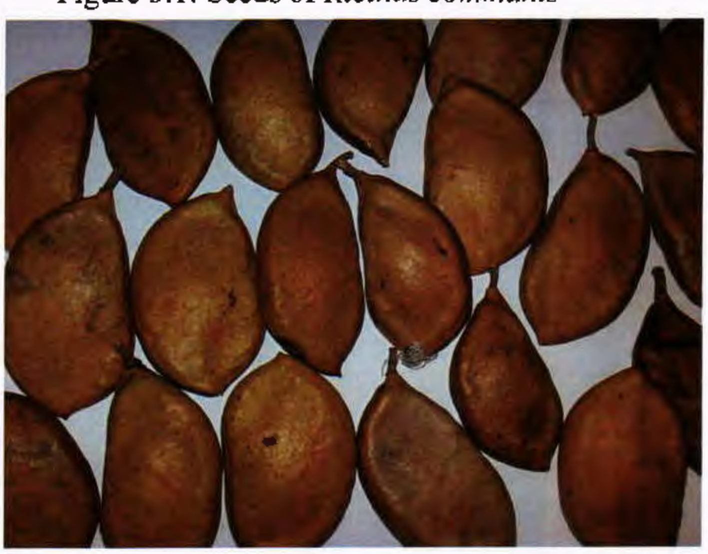

Figure 3.2: Seeds of *Pongamia pinnata*

Figure 3.3: Seeds of *Azadirachta indica*

3.1.3 Sample collection

Two samples namely *Ricinus communis* (Castor, local name: Arind) and *Pongmaia pinnata* (Sukh chain) seeds were collected from Chountra region near Wandala river of district Rawalpindi. *Azadirachta indica* (Neem) is not found in local areas and had to be purchased from agricultural market. These samples are widely available and are used for different purposes besides biodiesel the oils form these plants are available and used for medicinal purposes.

3.2 Methods

3.2.1 Scheme of study

This research was carried out according to the following diagram shown below.

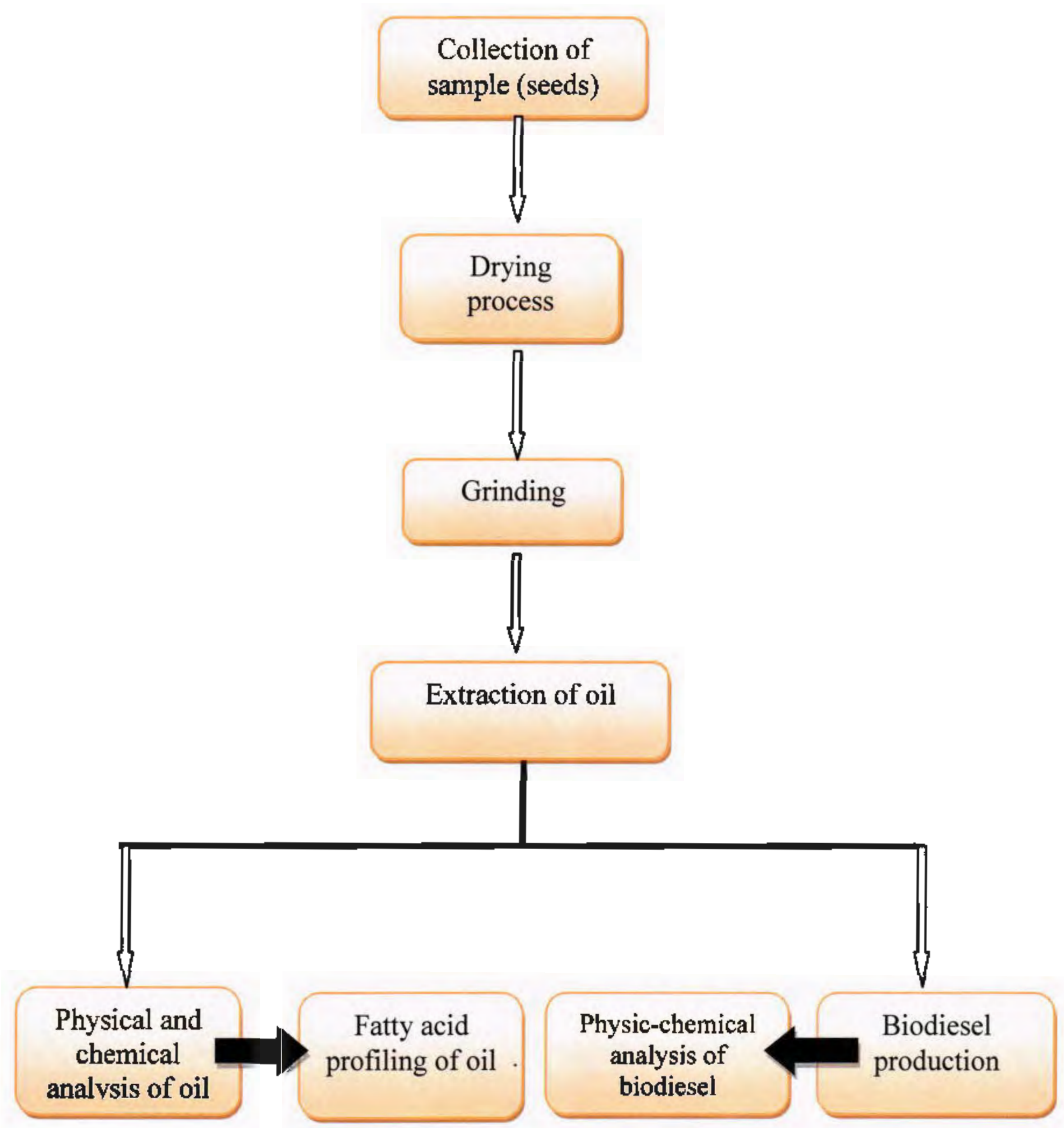

Figure 3.4: Schematic representation of overall objectives

3.2.2 Desiccation of samples

The desiccation of samples was performed by proliferation on the ground. This resulted in dehydration of seeds. The samples were shadow dried which resulted in splitting of seed coat. The seeds were again subjected to drying in an oven at 50^0C for 5 to 6 hours, the moisture content was reduced and a constant weight was obtained (Sousa *et al.*, 2010).

3.2.3 Crushing of seeds

The samples were loaded into a crushing machine which pulverized the samples. This crushed material was placed in refrigerator. This powdered material produced oil via Soxhlet apparatus.

3.2.4 Extraction of oils by Soxhlet Apparatus

Powdered samples were loaded one at a time inside thimble. The sample size was twenty grams. This apparatus was fitted with condenser which converts the vapours of the solvent (hexane) into liquid form. About 1/3rd litre of volatile liquid was put inside a flask, to condense the solvent water supply must be present to convert the vapours of solvents into the liquid form. About 50^{0}C heat is necessary for the liquids to back flow. This caused the liquids to turn into vapours and these vapours were converted into liquid state via condenser. In this way the samples absorbed the volatile liquids. This cycle was repeated many times until the liquids showed that the oil from samples has been extracted. Appearance of solvents changed due to presence of oil in it. This extract was channeled into another flask for collecting the sample containing oil. The solvent was removed from oil via rotary evaporator and pressure. The mass of oil produced was measured via digital balance and recorded after removing all the impurities by heating at 55^{0}C. The whole process took one day to complete the oil extracted was later analyzed for physical and chemical properties (Azadmard *et al.*, 2005).

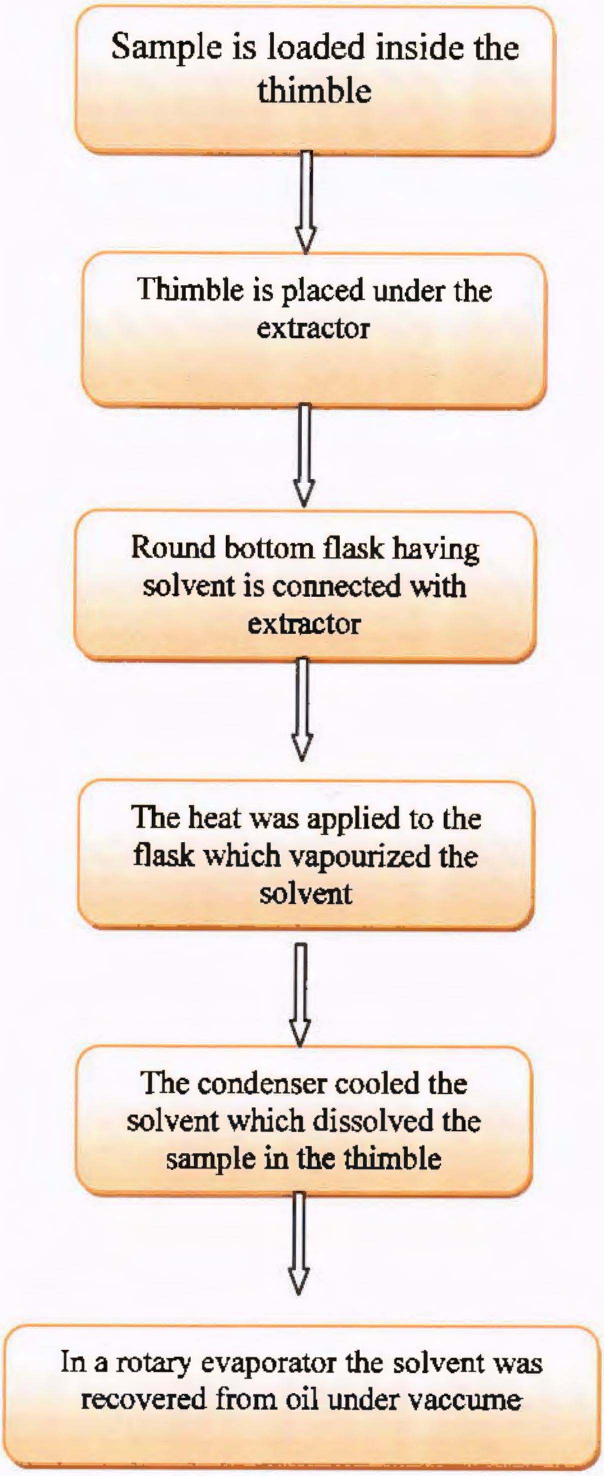

Figure 3.5: Working process of Soxhlet

3.2.5 Mechanical production of oils

The oil was produced by mechanical method also. The sample seeds were desiccated under natural light when water content of seeds was abated these were again picked up from desiccating area and placed inside polythene bags. This method removed outer covering of the seeds which is necessary for removal of unsaturated fat from seeds. Mechanized extraction was carried out in a machine which pressed the seeds and extracts maximum amount of oil. The remaining part of the seed from which oil had been extracted can be used as cattle feed. This process can be repeated many times for maximum extraction. The extraction of all the samples was performed in the same way. During the extraction process several unwanted materials like chunks of seeds and dust are mixed with oil. To remove these materials filtration process is performed. The oil is passed through filter paper about 5 times until all the fragments (Atabani and Silitonga 2013)

Figure 3.6: Schematic representation of Mechanical method

3.2.6 Physical and chemical characterization of oils

The oils were converted into biodiesel and characterized via different methods including physical methods and chemical methods.

3.2.6.1 Specific Gravity ASTM (D 1298)

Mass of 25ml glass specific gravity bottle was calculated using digital weight machine. This weight is shown as W_E mass of this bottle was weighed while it was filled with water this is recorded as W_W then mass of bottle again was measured while it contained sample oil this mass was recorded as W_S. S.G was found out by subtracting W_E from W_S.

then dividing it by (W_W-W_E). This procedure was performed at 20°C (Venkatesan and Vikram 2012)

Figure 3.7: Specific gravity of *Ricinus communis* oil

Figure 3.8: Specific gravity of *Azadirachta indica* oil

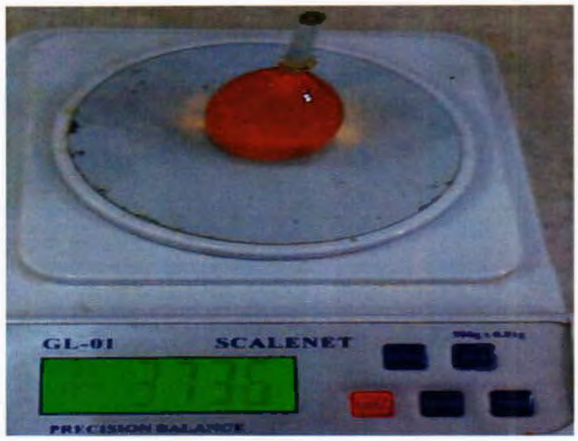

Figure 3.9: Specific gravity of *Pongamia pinnata* oil

3.2.6.2 Kinematic viscosity ASTM (D 445)

To determine the viscosity of oil the instrument used was Ostwald's viscometer. The instrument used was subjected to water bath for 25min. The sample was added to the viscometer when the water bath temperature reached 40^0C. The sample was sucked between the calibration marks. The time required for the oil to flow from one mark to other w0as calculated via using stopwatch. To obtain constant value the process was repeated about 3 times. The value was found by adding the values, dividing it by 3 then multiplied it with viscometer constant to find kinematic viscosity of given oil (Indhumathi *et al.*, 2014)

3.2.6.3 Determination of Acid value ASTM (D 664)

Acid value was determined by pouring of oil or biodiesel into conical flask taking 5ml of sample and 0.2 grams of fat solvent i.e 1:1 solution of ethanol and diethyl ether. The blank was also taken containing only fat solvent and phenolphthalein. 0.1M KOH was taken in beurette and analysis was performed for both sample and blank. The end point was reached when pink colour appeared and persisted for more than 10 sec (Akubugwo and Chinyere 2008). Following formula was used to calculate acid value;

Acid Value = 56.1 x Normality of Base x Volume of Base (ml) / Weight of sample

3.2.6.4 Calculation of Saponification Value ASTM (D 5558)

This value was found out by measuring 1 gram of oil or biodiesel and poured in a conical flask about 12.5 ml of 0.5 m/m solution of KOH was also dissolved into the same apparatus. The fat was converted to soap and liquid portion was separated and titrated with HCl having normality of 0.5. Same procedure was performed without oil in it. The formula used in this method is shown below (Akubugwo and Chinyere 2008)

Sap. Value = 56.1x Normality of HCl x Volume of HCl (test – blank)/ Weight of sample

3.2.6.5 Calculation of Iodine Value ASTM (D 1510)

1 gram of oil was measured in a flask. About 50ml of $CHCl_3$ was also added in the same flask. Wijs solution about 125 ml was taken out of stock solution and added to the flask. This apparatus was put in a place without light for half an hour and it was shaken constantly. KI solution was also added after words by pipetting out 50ml of it. This mixture was titrated with Sodium thiosulphate ($Na_2S_2O_3$) the end point was reached when yellow color disappeared and solution turned color less. Starch solution was used as in (Akubugwo and Chinyere 2008)

I_2 value = 12.69 x Normality of $Na_2S_2O_3$ x Volume of $Na_2S_2O_3$ (test -blank)/ sample weight

3.2.6.6 Free Fatty Acid percentage

This is calculated by dividing acid value with 2 the FFA is usually half of the acid number.

3.2.7 GC analysis of plant oils

Oil samples were subjected to GC analysis. Perkin Elmer Clarius 500 series was used for this purpose. Column HP Capillary: (60m)(0.25mm)(0.25um), this column was initially given the 60^0C command for about 6min, the temperature was raised afterwards to 110^0C. After 20 min the temperature was again raised to 200^0C in a constant manner 5 centigrade per minute. Similarly the temperature again rose to 220^0C. The final temperature of detector reached at 275°C. Hydrogen gas was injected at 45 psi while gaining velocity 39cm/s, the flow rate of column reached 2.4cc/s, the sample volume was 1 x 10^{-6}L. FFA profile was obtained for different acids present in the oil (Khobchandani *et al.*, 2010).

3.2.8 Fatty acid methyl esters synthesis

3.2.8.1 Transesterification of Fatty Acids

The fresh oil produced by different methods still contains many chemically active species and compounds. For conversion into biodiesel the presence of moisture is inadmissible. For this synthesis to run smoothly pure oil is required without any unwanted particle. The process of transesterification is carried out by using alkali base to speed up chemical reaction. The volume of basic ROH is kept 6 times more than the oil sample. A basic solution of either CH_3OH or C_2H_5OH or any other suitable alcohol was prepared having solute concentration of $1/100^{th}$ of total solution. This reaction must be moisture free. In the present work methyl and sodium hydroxide were utilized for the biodiesel synthesis. The basic alcohol solution was put in a flask along with oil and was placed over magnetic stirrer. The oil was first heated in a water bath at 70^0C then it was allowed into the reaction. The reaction was carried out for 3hours and then it was stopped cooled and poured into the separating funnel. The seoparating funnel was left for about 12hours and 12hours two layers were formed. Upper layer contained methyl esters and lower layer had glycerine and along with impurities (Antolin and Tinaut 2002).

3.2.8.2 Removal of unreacted species

H_3PO_4 was added to reaction mixture to get rid of any alkali base ion still left in the solution unbound to any other chemical ion thus free to react. This step forms salts which are dissolved in water when it is introduced for washing. The soap formation takes place when base reacts with fat molecule. This affects the production of biodiesel negatively in fact whole of it is wasted (Van-Gerpen 2005).

3.2.8.3 Washing of Biodiesel

The biodiesel produced has many unreacted species like alcohol, catalyst and glycerine molecules. This requires washing with water several times until a clear layer is visible. The moisture then is removed via placing it in an oven at 40 ^{0}C. Further drying is carried out with sodium sulphate followed by filtration (Rashid *et al.*, 2010).

3.2.8.4 Scheme for biodiesel production

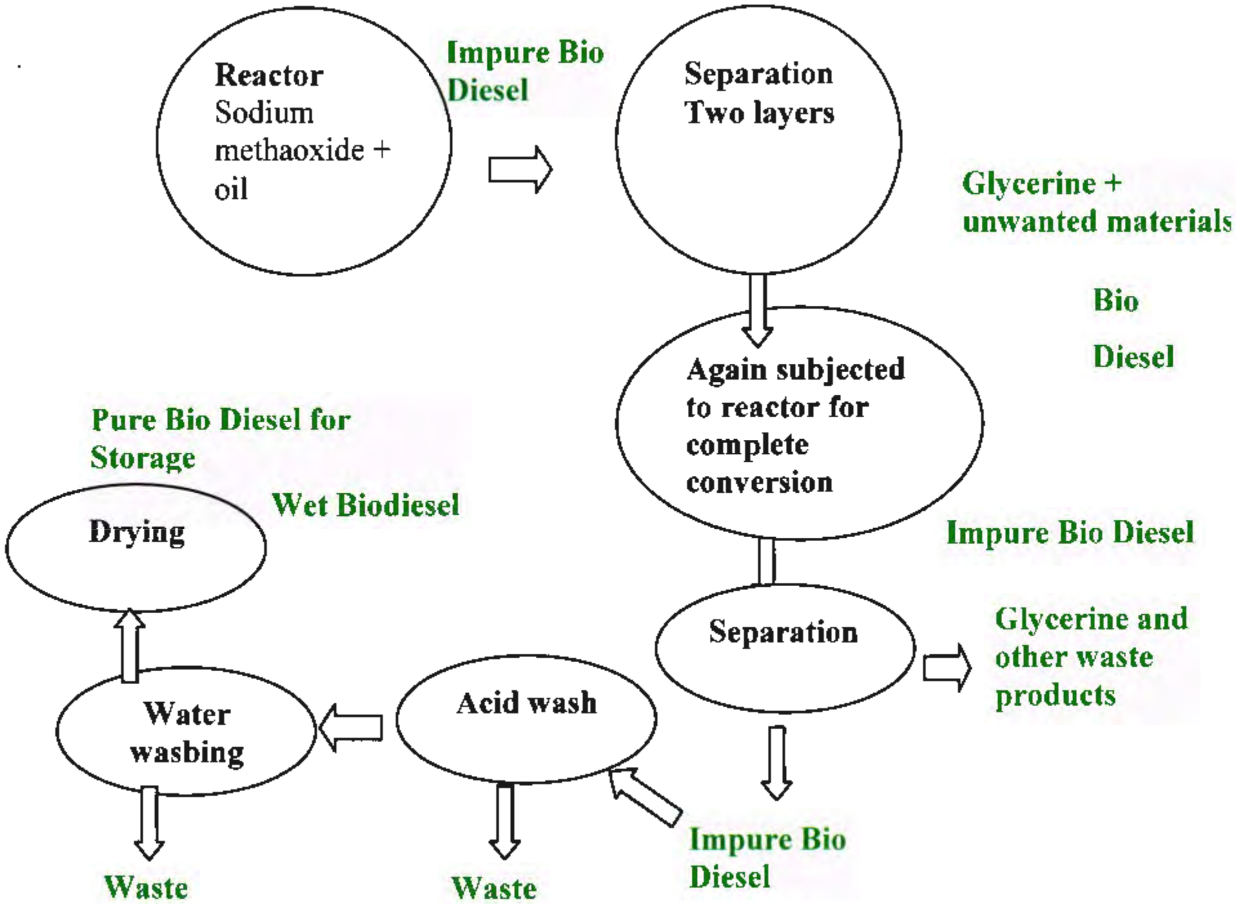

Figure 3.10: Scheme for biodiesel production

3.2.9 Characterization of biodiesel

The biodiesel produced was again characterized for physical and chemical parameters.

3.2.9.1 Physical parameters

The biodiesel produced was again characterized for physical and chemical parameters. Physical properties such as specific gravity and kinematic viscosity are calculated via specific methods of ASTM (1992).

3.2.9.1.1 Specific gravity (D 1298)

Mass of 25ml glass density bottle was calculated using digital weight machine. This weight is shown as W_E mass of this bottle was weighed while it was filled with water this is recorded as W_W then weight of bottle again was measured while it contained biodiesel this weight was recorded as W_S. S.G was found out by subtracting W_E from W_S. then dividing it by (W_W-W_E). This procedure was performed at 20°C (Venkatesan and Vikram 2012)

Figure 3.11: Specific gravity of *Ricinus communis* biodiesel

Figure 3.12: Specific gravity of *Azadirachta indica* biodiesel

Figure 3.13: Specific gravity of *Pongamia pinnata* biodiesel

3.2.9.1.2 Kinematic viscosity (D 445)

A viscometer was inserted into a water bath with a set temperature and left for 30minutes. The biodiesel was added to the viscometer and allowed to remain in the bath as long as it reaches the test thermometer. The sample was allowed to flow freely and the time required for the meniscus to pass from the first to the second timing mark is taken using a stop watch. The procedure is repeated a number of times and the average value are taken which is then multiplied with the viscometer calibration to give the kinematic viscosity (Indhumathi *et al.*, 2014).

Figure 3.14: Kinematic viscosity of *Ricinus communis* biodiesel

Figure 3.15: Kinematic viscosity of *Azadirachta indica* biodiesel

Figure 3.16: Kinematic viscosity of *Pongamia pinnata* biodiesel

3.2.9.2 Chemical parameters

Acid value and iodine value were determined via methods described by (Akubugwo and Chinyere 2008) according to ASTM D 664 for acid value and ASTM D 1510 for iodine value.

3.2.9.2.1 Acid value for biodiesel ASTM (D 664)

Acid value was determined by pouring of biodiesel into conical flask taking 5ml of sample and 0.2 grams of fat solvent i.e 1:1 solution of ethanol and diethyl ether. The blank was also taken containing only fat solvent and indicator. 0.1M KOH was taken in burette and analysis was performed for both sample and blank. The end point was reached when pink color appeared and persisted for more than 10 sec (Akubugwo and Chinyere 2008). Following formula was used to calculate acid value;

Acid Value = 56.1 x Normality of Base x Volume of Base (ml) / Weight of sample

3.2.9.2.2 Iodine value for biodiesel ASTM (D 1510)

One gram of biodiesel was measured in a flask. About 50ml of $CHCl_3$ was also added in the same flask. Wijs solution about 125 ml was taken out of stock solution and added to the flask. This apparatus was put in a place without light for half an hour and it was shaken constantly. KI solution was also added after words by pipetting out 50ml of it. This mixture was titrated with sodium thiosulphate the end point was reached when yellow color disappeared and solution turned color less. Starch solution was used as in (Akubugwo and Chinyere 2008)

I_2 value = 12.69 x Normality of $Na_2S_2O_3$ x Volume of $Na_2S_2O_3$ (test -blank)/ sample weight

RESULTS

RESULTS

Methods specified by ASTM were used determining the properties of oils and biodiesels (Storer and Cornillot 1992). The sample selected for this study produce nonedible oils not involved in the utilization of food. For optimum use in diesel engines the oils are converted into biodiesel. The characters which are suitable for maximum performance are studied by carrying out different chemical tests and analysis. The amount of acid radicals, unsaturation and soap formation determined whether quality biodiesel is produced or not.

4.1 Production of oil

The seeds used in this study were cleaned first. For quelling the seeds a machine connected with motor was used. The seeds for each sample were 5kg. According to the literature the percentage of oil production for castor was <50%. In this study the percentage yield for Castor (*Ricinus communis*) is 43%. Similarly *Pongamia pinnata* produced 39% oil which agrees well with the values found in the literature. *Azadirachta indica* oil extraction showed that it produced less amount of oil than both the samples and produced 28% oil from its seeds as indicate in table (4.1) and figure (4.1).

Table 4.1: Oil yield of three different plants

Plant	Weight of sample	Oil content	Percentage yield
Ricinus communis	5kg	2.15L	43%
Azadirachta indica	5kg	1.4L	28%
Pongamia pinnata	5kg	1.9L	39%

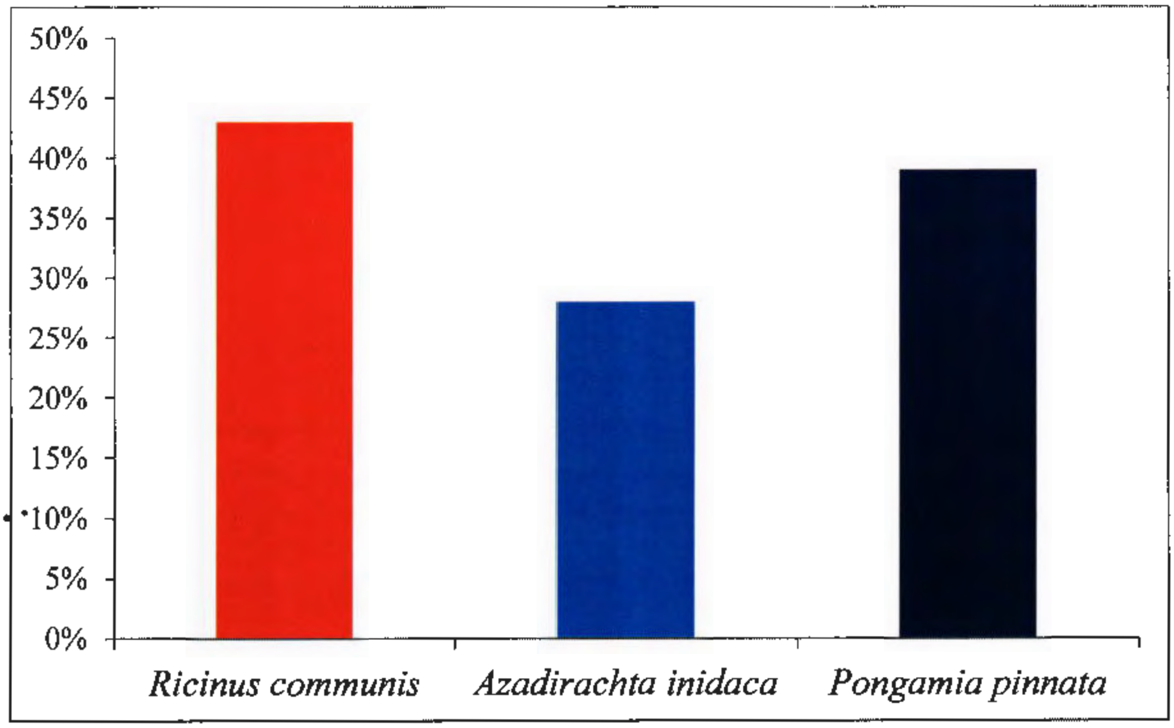

Figure 4.1: Graph indicate the percentage of oil yield by mechanical method

4.2 Production of oils via Soxhlet apparatus

The oil from samples *Ricinus communis*, *Azadirachta indica* and *Pongamia pinnata* was extracted using soxhlet apparatus. The yield is given in the table (4.2) and figure (4.2) below.

Table 4.2: Percentage of oil yield by Soxhlet

Plant	Weight of sample	Oil content	Percentage yield
Ricinus communis	20g	9 grams	45%
Azadirachta inidaca	20g	6.4 grams	32%
Pongamia pinnata	20g	8.2 grams	40%

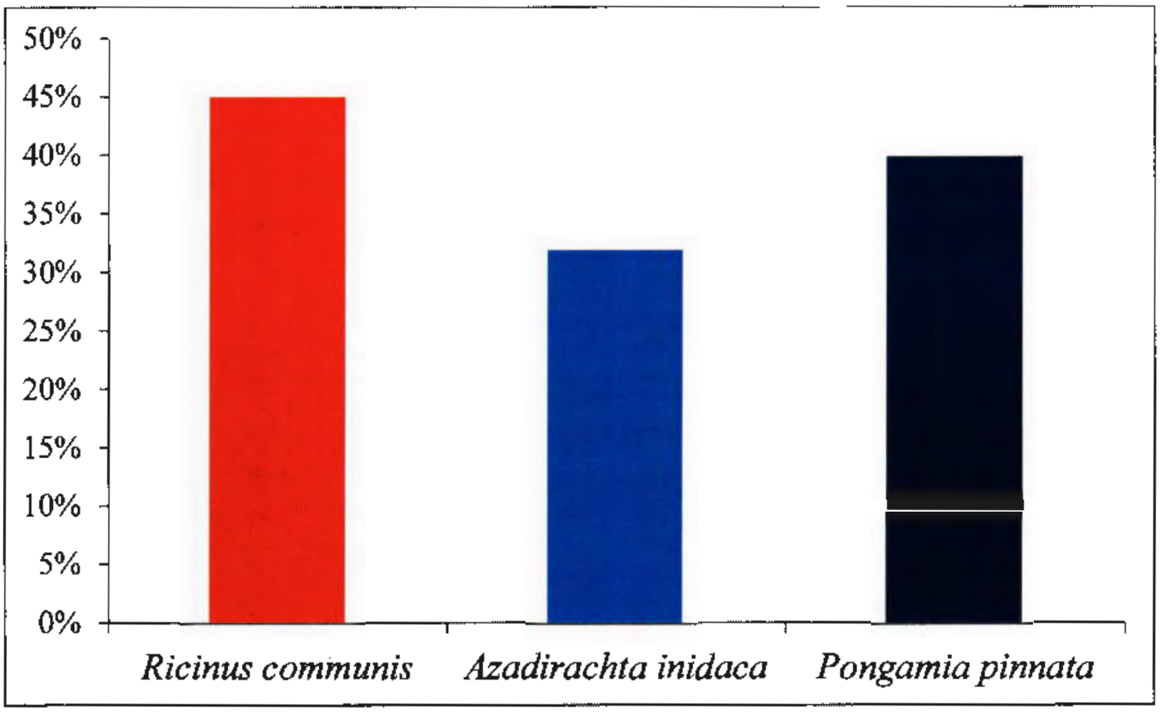

Figure 4.2: Graph indicate the percentage of oil yield by Soxhlet method

4.3 Physical properties of plant oils

Characters such as specific gravity and kinematic viscosities are indicated inside table (4.3). *Ricinus communis* showed the highest specific gravity value and also kinematic viscosity these are 970kg.m^{-3} and 65cSt respectively. On the other hand *Pongamia pinnata* showed slightly lower S.G value 910kg.m^{-3} and its viscosity is calculated 11.49cSt. *Azadirachta indica* has less Specific gravity than both the oils 900kg.m^{-3} its Kinematic viscosity is also lowers than other oils which is 11.4cSt according to this study.

4.4 Chemical properties of oils

To determine chemical properties the presence of fatty acids, unsaturated molecules and ability of oils to form soap is estimated. This is performed via methods described by ASTM. Acid value test was performed first which was followed by I_2 and soap value of oils.

Ricinus communis has shown that it has highest acid value and I_2 value and lowest saponifcaiton value these values are 10.4 mg.g^{-1} and 84.5mgI_2.g^{-1} hut Saponification value 178.3 mg.g^{-1}.

Azadirachta indica showed highest Saponification value and lowest unsaturation value these are 198 $mg.g^{-1}$ and 69.77 $mgI_2.g^{-1}$ respectively. Its acid value was calculated to be 7.54 $mg.g^{-1}$. Lowest acid value is shown by *Pongamia pinnata* which is 4.8 $mgI_2.g^{-1}$. Its iodine value and Saponification are 81 $mgI_2.g^{-1}$ and 189 $mg.g^{-1}$ respectively. This study agrees with the work carried out in the past.

4.5 Fatty acid profiling

Various Fatty acid from different oils were analyzed through gas chromatography *Ricinus communis* has high content of ricinoleic acid 85.4% linoleic acid 5.6%, stearic acid 2.4%, palmitic acid 1.2%, linolenic acid 1.1%. *Azadirachta indica* oil indicated the percentages of oleic acid 44.9%, stearic acid 18.5% and palmitic acid 16.8%, these were in considerable amount while ricinoleic acid and linolenic acid were 0.4 ± 0.1 and 0.4 ± 0.2 respectively. In *Pongamia pinnata* oil the values for oleic acid 48.8%, linoleic acid is 16.8%, palmitic acid 13.6% and linolenic acid 11.2% were observed.

Ricinus communis contain higher concentration of ricinoleic acid 85.4% . Oleic acid was higher in percentage at 44.9% in *Azadirachta indica* while linolenic acid was least at 0.4%. *Pongamia pinnata* oil showed oleic acid 48.8% while ricinoleic acid is least in percentage. Among the three oils *Ricinus communis* is lower in saturated acids at 3.6%. *Azadirachta indica* showed higher amount of saturated acids 35.3% while *Pongmaia pinnata* is intermediate at 21%. Higher amount of monounsaturated fatty acids are found in *Ricinus communis* 88.5% least amount of monounsaturated fatty acids are found in *Azadirachta indica* 45.3%. *Pongamia pinnata* has 28% poly unsaturated fatty acids, *Azadirachta indica* has 19.3% poly unsaturated fatty acid.

4.6 Production of biodiesel

Yield of biodiesel from all the samples is shown in following graph. The conversion of oil into fatty acid methyl esters produces glycerin and biodiesel.

4.7 Biodiesel characterization

Biodiesel of *Ricinus communis* showed again higher value of specific gravity than other biodiesels which is $930kg.m^{-3}$. Its kinematic viscosity was shown at 5.97cSt both these values are comparable to ASTM values. Acid values and iodine values are also dropped to $1.2mg.g^{-1}$ and about $80mgI_2.g^{-1}$ respectively.

Pongmia pinnata biodiesel showed that its iodine value is higher than other oil which in this case is 86.6 $mgI_2.g^{-1}$. The acid value of this oil is well within the range specified by standards of normal and ASTM biodiesel. The viscosity and specific gravity of this oil is also within range of standards as shown in table also.

Lowest S.G value for biodiesel and Iodine value is shown by *Azadirachta indica*. 860$kg.m^{-3}$ and iodine value approximately 68 $mgI_2.g^{-1}$ was calculated in this study. Acid value and kinematic viscosity are within range of ASTM which is 4.9 $mm^2.s^{-1}$.

Table 4.3: Physical and chemical properties of oils

Physical and chemical properties	Method used ASTM	*Ricinus communis*	*Azadirachta indica*	*Pongamia pinnata*
Specific gravity($kg.m^{-3}$)	D 1298	970	900	910
Kinematic viscosity (cSt)	D 445	65	11.4	11.49
Acid Value ($mgKOH.g^{-1}$)	D 664	10.4	7.64	4.77
Iodine value($mg.I_2.g^{-1}$)	D 1510	84.5	69.77	80.56
Sap. value ($mgKOH.g^{-1}$)	D 5558	178.3	198.5	188.9

Table 4.4: Percentage of biodiesel yield from three different plants

Name of sample	Amount of solution Sodium methaoxide	Volume of oil used	Percentage yield of FAME
Ricinus communis	240ml	40ml	90%
Azadirachta inidaca	240ml	40ml	85%
Pongamia pinnata	240ml	40ml	78%

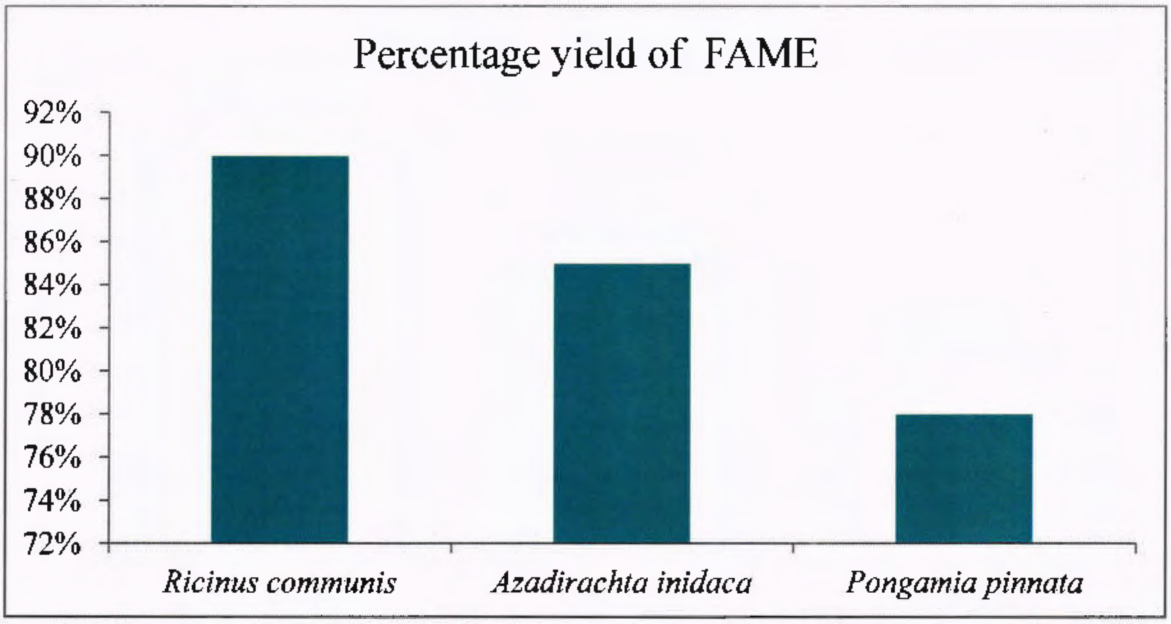

Figure 4.3: Percentage yield of biodiesel (FAME)

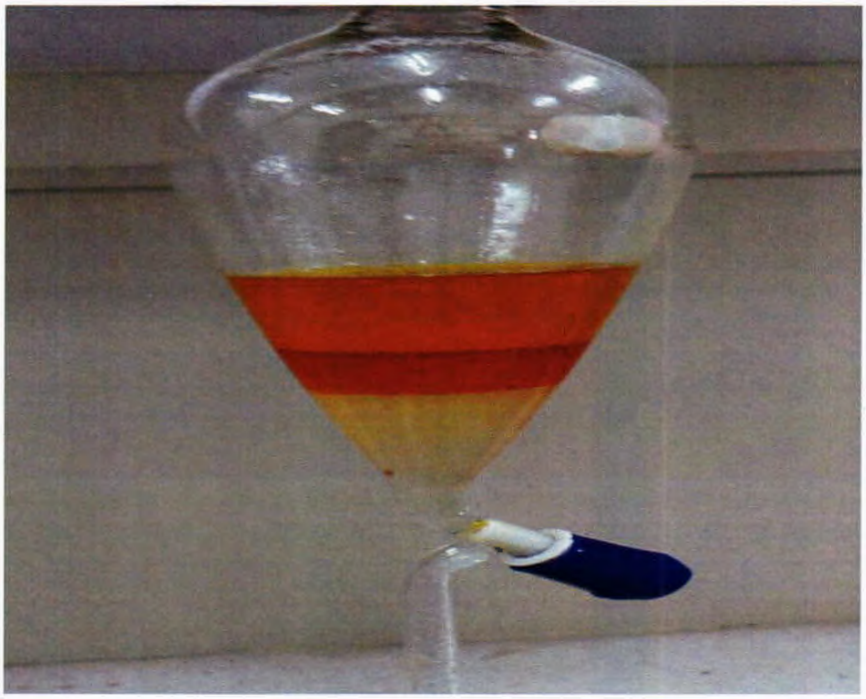

Figure 4.4: *Ricinus communis* biodiesel separation

Figure 4.5: *Azadirachta indica* biodiesel separation

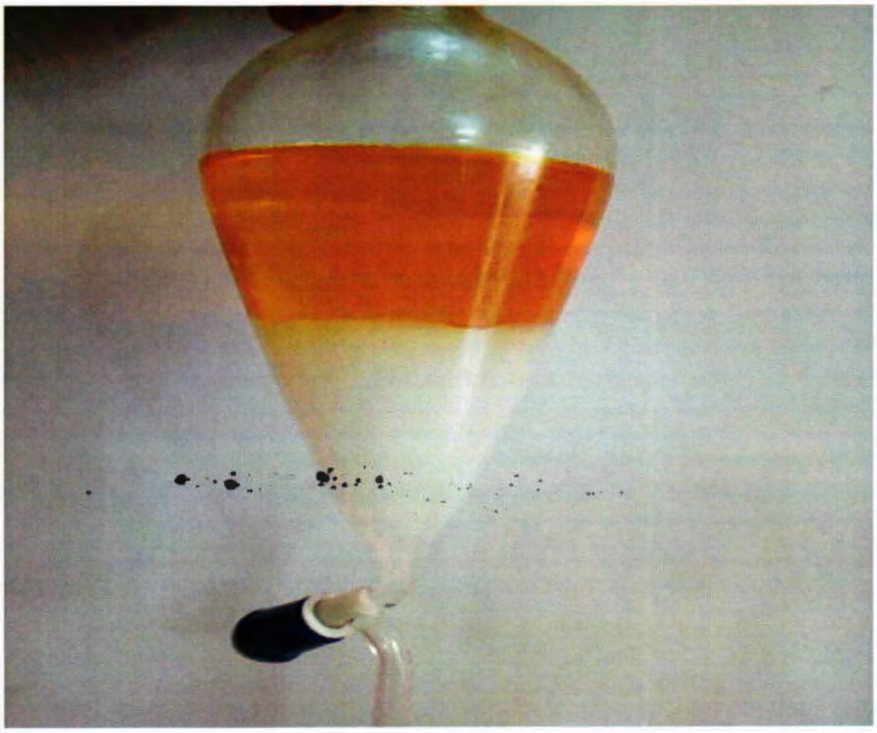

Figure 4.6: *Pongamia pinnata* biodiesel separation

Table 4.5: Physical and chemical properties of biodiesel

	Method used	ASTM standard value for diesel	ASTM standard value for biodiesel	*Ricinus communis*	*Azadirachta indica*	*Pongamia pinnata*
S.G ($kg.m^{-3}$)	D 1298	950	890	930	860	890
Kinematic viscosity (cSt)	D 445	1.9- 4.1	1.9 - 6	5.97	4.99	5.6
Acid Value $mgKOH.g^{-1}$	D 664	0.50	0.80	1.2	0.78	0.54
Iodine value $mgI_2.g^{-1}$	D 1510		82-98	80.8	67.6	86.6

Table 4.6: Fatty acid profiling of oils from Gas Chromatography

Fatty acids	*Ricinus communis*	*Azadirachta inidca*	*Pongamaia Pinnata*
Palmitic acid $_{(C16:0)}$	1.2 ± 0.1	16.8 ± 0.2	13.4 ± 0.1
Stearic acid $_{(C18:0)}$	2.4 ± 0.3	18.5 ± 0.1	7.4 ± 0.2
Oleic acid $_{(C18:1)}$	3.1 ± 0.0	44.9 ± 0.3	48.8 ± 0.1
Ricinoleic acid $_{(C18:1)}$	85.4± 0.1	0.4 ± 0.1	1.9±0.2
Linoleic acid$_{(C18:2)}$	5.6 ± 0.2	18.7 ± 0.1	17.1 ± 0.4
Linolenic acid $_{(C18:3)}$	1.1 ± 0.1	0.4 ± 0.2	11.1 ± 0.1
Arachidic acid $_{(C20:0)}$	1.2±0.2	0.2 ± 0.1	nd**

*Values presented Mean ± standard deviation

** nd = not detected

Table 4.7: Percentage of saturated and unsaturated fatty acids

Types of fatty acids	*Ricinus communis*	*Azadirachta indica*	*Pongamia pinnata*
(SFA)[a]	3.6%	35.3%	21%
(MUFA)[b]	88.5%	45.3%	50.7%
(PUFA)[c]	7.9%	19.3%	28%
others	0.1%	0.1%	0.3%

a=(SFA= saturated fatty acid), b= (MUFA= mono unsaturated fatty acid), c=(PUFA =poly unsaturated fatty acid)

DISCUSSION

DISCUSSION

Globally energy demand is increasing day by day. This energy demand is met by fossil fuels which is harmful for environment and economy. The society is suffering from both sides from bad environmental conditions and higher prices of fuels. There is also a danger of losing fossil fuel for good. To compensate for the loss of fossil fuels the society must find alternative resources to cope with depleting energy reserves and degrading environment. The new resource should be such that it must not interfere with basic need of humans' i.e food. Non edible oil produced by many plants is considered as one of the major source of fuel production. It eliminates the competition between food crops and non food crops.

The plants used in this study are *Ricinus communis, Azadirachta indica and Pongamia pinnata.* The oil obtained from these plants are used as medicines and for the production of biodiesel. The biodiesel produced can be utilized in diesel engines of motor vehicles and electricity generators.

The research has been conducted on non edible plant oils to find out percentage yield of each oil physical and chemical parameters and conversion into biodiesel. The biodiesel produced was again checked for the same characteristics and percentage yield. In this study three plants were selected for characterization of non edible oils *Ricinus communis, Azadirachta indica* and *Pongamia pinnata* all plants are found locally. These plants produce non edible plant oils having high acid value. Different methods were utilized to determine physico chemical properties of oils such as specific gravity, kinematic viscosity, acid value, iodine value and saponification value.

The samples were utilized for the extraction of oils via mechanical and soxhlet apparatus. The mechanical methods performed for *Ricinus communis* yielded 43% oil *Azadirachta indica* yielded 28% and *Pongamia pinnata* 39%. The reported value for *Ricinus communis* by Gokdogan and Eryilmaz (2015) is about 50% which is higher than our reported vlaue. Kaushik and Vir (2000) reported that *Azadirachta indica* yield upto 30% oil from seeds these finding are in agreement with our study. Gawali and Wagh, 2015 reported 40% oil content from *Pongamia pinnata* seeds this too is in accordance to our values.

The soxhlet apparatus yield for *Ricinus communis* 45%, *Azadirachta indica* 32% and for *Pongamia pinnata* 40%. Nangbes *et al* (2013) extracted oil from Ricinus communis and reported value of 48% yield. This showed that our yield is comparable with the literature. Awolu and Obafaye (2013) found that *Azadirachta indica* oil gave yield of 49% which is higher than reported in our study this may be due to the fact that our seeds may have less oil content due to climatic conditions in local area. Bodabe and Khyde (2012) analyzed the *Pongamia pinnata* oil and performed different types of extraction and found that Soxhlet apparatus gave better yield than mechanical and cold percolation method they observed value of 31% which is less than obtained in this study. This may be due to the fact that oil percentage in seeds is higher in this region and produce better percentage of oil.

Fatty acid profiling of *Ricinus communis* oil was assessed with the help of gas chromatography and it was found that ricinoleic acid is 85.4%, linoleic acid is 5.6% and oleic acid is 3.1 %. The presence of ricinoleic acid was in accordance with the reported value while linoleic acid and oleic acid was slighlty less than reported vlaue in the literature. Percentage of ricinoleic acid linoleic acid is quite comparable Salimon and Noor (2010) while saturated fatty acid content showed some variations.

Azadirachta indica oil was subjected to GC analysis it was determined that it is high in sturated fatty acid i.e C16:0 and C18:0 these are 35.3% which is 10% higher than reported by Djenontin *et al.,* (2012) the rest of the fatty acids are unsturated having 45.3% monounsturated and 19.3% poly unsturated fatty acid. The above cited scientist anlyzed *Azadirahcta indica* oil and compared it to other oils they reported the presence of oleic acid at 25% while this amount is lower than observed in the present study. The percentage of palmitic acid is much closer to reported in the literature. The amount of stearic acid is also found to be higher than present in the cited literatuere.

Meher and Dharmagadda (2006) determined fatty acid profiling of *Pongamia pinnata* oil and it was found that oleic acid is present in higher amount which is indicated by our study as well. The present study showed oleic acid 48.8% this vlaue along with percentages of palmitic acid and linoleic acid compare well with cited literature. In our study the presence of saturated fatty acid is 21% this is less than reported in the literature which is about 26%.

The densities of *Ricinus communis*, *Azadirachta indica* and *Pongamia pinnata* oil were measured and recorded to be 970kg.m^{-3} 900kg.m^{-3} and 910kg.m^{-3} respectively. The density value of castor oil is in accordance with ASTM standard value for quality castor oil which is 957- 961 kg.m^{-3}. Al-Harbawy and Al-Mallah (2014) who reported that *Ricinus communis* has density value 960 kg.m^{-3} which confirm our research about finding density value. Ndana *et al.,* (2010) have conducted a research on plants of local origin for the biodiesel production and for determination of physical and chemical properties found density of neem oil as 930kg.m^{-3}. Venkatesan and Vikram (2012) analyzed the Pongamia methyl esters and its use in diesel engines as also a part of diesel fuel in blended form. They reported that this oil shows density 940kg.m^{-3} which is slightly more than our reported value.

The resistance to the flow of liquid is viscosity. A biodiesel having high value of viscosity is not suitable it will not flow properly. In the present study the viscosity of three oil was measured the determined values regarding each oil are recorded as *Ricinus communis* (Castor oil) 65cSt, *Azadirachta indica* (Neem oil) 11.40cSt and of *Pongamia pinnata* (kranja) is 11.49cSt. Gokdogan and Erylmaz (2015) studied castor oil and its blends. They found out that castor oil has viscosity 241.46cSt. This shows that our value is less than values reported in the literature may be due to the fact that it may have been stored in high temperature environment or lab conditions. This high viscosity must be brought down via transesterification reaction for utilising in diesel engine. Aransiola and Betiku (2012) in their study on Neem oil for biodiesel production reported neem oil viscosity as 43.75cSt. These findings suggest that viscosity of oil is dependent on environmental conditions such as high temperature and storage conditions and lab environment. Bobade and Khyade 2012 studied properties of *Pongmia* oil for biofuel production and reported value of 40.2 cSt for Pongamia oil viscosity.

Acid vlaues is measure of acids prsent in the crude oil if the amount of acid value is high it will produce soap and deactivate the catalyst. Vegetable oils with high acid value are classified as inedible while those with low acid value are classified as edible oil (Leung and Guo

2006). The oils used in this study are non edible because of high acid value and thus do not compete with food crops their acid values are determined using standard methods and techniques. Acid value of *Ricinus communis* in this study is determined which was 10.4 $mg.g^{-1}$. Salimon and Noor (2010) analyzed Malaysian castor oil and studied its physical and chemical properties and types of acids present in this oil. In their research they find that Castor oil found in their region showed acid value of 4.9 $mg.g^{-1}$ high acid value in our study suggest that lipase enzyme cleaved esters in the seed which produced more acid value. In case of Neem oil (*Azadirachta indica*) when subjected to acid value test and it was calculated as 7.64$mg.g^{-1}$. Abdulkadir and Adisa (2014) in their research on combustion analysis of *Azadirachta indica* its extraction of oil and properties related to physical and chemical aspects determined acid value for 6.77 $mg.g^{-1}$, this slight variation of results suggest that temperature differences in areas is responsible for the activation of lipase enzyme which is cause of more acid value.

Similarly acid value of *Pongamia* crude oil was determined. . In this study the acid value was measured as 4.77mgKOH/g. Meher and Dharnagadda (2006) in their research on role of catalyst in biodiesel conversion process and optimization of conditions for the reaction. They calculated the acid value of Pongamia oil as 5.06 mgKOH/g.

Iodine value is level of unsaturation of oil. I.V of three oils was measured and compared with the values found in the literature. In this study *Ricinus communis* oil was analyzed for the iodine value and calculated as 84.5$mgI_2.g^{-1}$. Asmare and Gabbiye (2014) found out iodine value while characterizing of biodiesel from castor oil for consumption in diesel engine. They found that IV for castor is 87.9$mgI_2.g^{-1}$. *Azadirachta indica* oil was also used for the analyzing iodine value the value found was 69.77 $mgI_2.g^{-1}$. Djibril and Lamine (2015) in their research calculated iodine value at 74.8$mgI_2.g^{-1}$. *Pongamia pinnata* oil was analyzed and the value found is 80.56 $mgI_2.g^{-1}$. Bodabe and Khyde (2012) in their research about *Pongamia* oil calculated iodine value as 87 $mgI_2.g^{-1}$.

Saponification value is tendency of plant oil to form soap during transesterification reaction. Higher values of soap indicate that it can be used for the production soap and detergents. In this study the Saponification values of *Ricinus communis* (Castor oil), *Azadirachta indica* (Neem oil) and *Pongamia pinnata* were calculated the value found were 178.3 $mg.g^{-1}$, 198.5 $mg.g^{-1}$ and 188.9 $mg.g^{-1}$ respectively. Asmare and Gabbiye (2014) carried out research on castor oil. They calculated Saponification value for castor oil as 185.3$mg.g^{-1}$. Aransiola and Betiku (2012) in their research about neem oil found that the saponfication value for neem was 199.8 $mg.g^{-1}$. Thiruvengadaravi *et al.*, (2012) found out the Saponification value for *Pongamia pinnata* as 190 $mg.g^{-1}$.

The ester yield for *Ricinus communis*, *Azadirachta indica* and *Pongamia pinnata* is 90%, 85% and 78% respectively. Asmare and Gabbiye (2014) reported 95% methyl esters yield more than calculated in this study. Sriramajayam (2016) reported about 98% methyl esters yield for *Azadirachta indica* higher than reported. Meher and Dharmagadda (2006) reported about 96% yield is also higer than reported value *Pongamia pinnata*. This suggest that our method produced more glycerine and rection was crried out with much more efficiency and at optimum conditions.

The plant oil was converted into biodiesel and analyzed for physical and chemical analysis. The Specific gravity of castor oil was measured as 930$kg.m^{-3}$ which is according to standard for petrodiesel while viscosity of this biodiesel is 5.97cSt which is in range of ASTM. Chemical parameters were also checked for biodiesel first of all acid value of castor oil biodiesel and iodine value were measured which is 1.2 $mg.g^{-1}$ and 80.8$mgI_2.g^{-1}$respectively. Berman and Nizri (2011) found density of castor biodiesel as 924$kg.m^{-3}$ and viscosity as 15.17cSt. Panwar and Shrirame (2010) reported value of castor biodiesel i.e 1.008 $mg.g^{-1}$.

Azadirachta indica oil was converted into biodiesel and analyzed. The density and viscosity of this biodiesel was calculated as 860$kg.m^{-3}$ and 4.99cSt respectively. Biodiesel of Neem oil was analyzed for chemical characteristics; in this regard acid value and iodine value were calculated. Acid value was 0.78$mg.g^{-1}$ and iodine value 67.6$mgI_2.g^{-1}$. Kinematic viscosity and acid value are under specific range of ASTM for biodiesel. Djibril and Lamine (2015) produced neem biodiesel and found physical characteristics such as specific gravity 970$kg.m^{-3}$ and viscosity as 35.5cSt. Sriramajayam (2016) found acid value of neem oil and iodine value 2.87$mg.g^{-1}$ and 77.87$mgI_2.g^{-1}$ respectively.

Density of *Pingamia pinnata* biodiesel in this study was found to be $890kg.m^{-3}$ and viscosity of biodiesel is 5.6cSt. S.G and kinematic viscosity for this biodiesel are in agreement for standard of ASTM. Acid value and iodine values are 0.54mg/g and 86.6mg/g respectively. Bobade and Khyade (2012) worked on Kranja oil methyl esters they calculated density $860kg.m^{-3}$, acid vlaue $0.46mg.g^{-1}$ and iodine value $91mgI_2.g^{-1}$.

CONCLUSION

CONCLUSION

The importance of biodiesel has increased in the present times due to its benefits. It is biodegradable thus environmentally friendly and has the commercial significance as well. Fossil fuels resources are being depleted and polluting the environment on global scale. There is a strong urge to find renewable ecofriendly and economically profitable fuel for benefit of society. Non edible oil could be a promising source for the production of biodiesel. Use of non edible oils avoids the problems associated with 1^{st} generation biodiesels. It can also be grown on lands not suited for normal food crops thus results in beneficial utilization of land and increase the aesthetic value of the land. Non edible oil used in this study are *Ricinus communis, Azadirachta indica and Pongamia pinnata* seeds.

The oils of these plants were extracted via mechanical and Soxhlet apparatus. The later gave better yield but it is costly and time consuming. The oil were subjected to physico-chemical analysis and then converted to biodiesel via transesterification reaction. Biodiesel was also analyzed for physical and chemical parameters. The comparison was drawn between reported values and standard values. It was found that many values fall within acceptable limits like yield of oil and transesterification compares well with values in the literature. Specific gravity of *Ricinus communis, Azadirachta indica* fall within acceptable limits. Kinematic viscosity and Acid values of *Azadirachta indica* and *Pongamia pinnata* biodiesel also defined and found according to the ASTM values.

This study showed that in the future biodiesel could become a valuable vehicle fuel although it cannot meet the demand being fulfilled by fossil fuel. If the cost of production of these energy crops could be brought down and methods associated with the extraction of oils are refined there is a huge chance to produce biodiesel on larger scale. The governments must provide subsidies to farmers and litigation for the use of land this could produce employment and enhance the economic activity in the nearby surroundings.

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