

# Experimental Investigation on Long term Storage Issues of Vegetable Oil and Biodiesel Fuel

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## Abstract

Energy is one of the major inputs for the economic development of any country. In the case of the developing countries, the energy sector assumes a critical importance in view of the ever-increasing energy needs requiring huge investments to meet them. For the developing country like India, economic growth is desirable and energy is essential for economic growth. The situation has led to the search for an alternative fuel, which should be not only sustainable but also environment friendly. For developing countries, fuels of bio-origin, such as alcohol, vegetable oils, biomass, biogas, synthetic fuels, etc. are becoming important. For the present work, Experimental investigations on long term storage issues of Vegetable Oil and Biodiesel fuel is carried out. Long-term storage study gives us a better understanding of the effect of the storage condition on the stability of biodiesel.

**Keywords:** Transesterification, Biodiesel, Jatropha, Pongamia, FFA (free fatty acid).

## 1. Introduction

The enormous growth of world population, increased technical development, and standard of living in the industrial nations has led to this intricate situation in the field of energy supply and demand. The prices of crude oil keep rising and fluctuating on a daily basis [1]. The situation has led to the search for an alternative fuel, which should be not only sustainable but also environment friendly. For developing countries, fuels of bio-origin, such as alcohol, vegetable oils, biomass, biogas, synthetic fuels, etc. are becoming important. The search for an alternative fuel, which promises a harmonious correlation with sustainable development, energy conservation, management, efficiency, and environmental preservation, has become highly pronounced in the present context [2]. Scientists around the world have explored several alternative energy resources, which have the potential to quench the ever-increasing energy thirst of today's population. These alternative energy resources are largely environment-friendly but they need to be evaluated on case-to-case basis for their advantages, disadvantages and specific applications. The main alternative fuels applied so far are oxygenates (alcohol, ether, etc), vegetable oil and its derivatives, gaseous fuels (hydrogen, liquefied petroleum gas etc), and coal derivatives. In Brazil, an infrastructure for production and supply of ethanol from sugarcane has led to the commercialization of over five million ethanol fuelled vehicles [3]. Diesel fuel is largely utilized in the transport, agriculture, commercial, domestic, and industrial sectors for the generation of power or mechanical energy and the substitu-

tion of even a small fraction of total consumption by alternative fuels will have a significant impact on the economy and the environment. The inventor of diesel engine, Rudolf Diesel, in 1885, used vegetable oils (peanut oil) as a diesel fuel for demonstration at the world exhibition in Paris. Speaking to the Engineering Society of St. Louis, Missouri, in 1912, Diesel said, "The use of vegetable oils for engine fuels may seem insignificant today, but such oil may become in course of time as important as petroleum and coal tar products of the present times". From the point of view of protecting the global environment and the concern for long term supplies of conventional diesel fuels, it becomes necessary to develop alternative fuels comparable with conventional fuels. Of the alternative fuels, biodiesel obtained from vegetable oils holds good promises as an eco-friendly alternative to diesel fuel [4]. The American Society for Testing and Materials (ASTM) defines biodiesel fuel as mono alkyl esters of long chain fatty acids derived from a renewable feed stock such as vegetable oil or animal fat [5]. The experimental results of various researchers support the use of biodiesel as a viable alternative to the diesel oil for use in the internal combustion engines. In recent years, systematic efforts were undertaken by many researchers to determine the suitability of vegetable oil and its derivatives as fuel or blend to the diesel [6, 7, 8, 9, and 10]. In view of environmental considerations, biodiesel is considered 'carbon neutral' because all the carbon dioxide released during consumption had been sequestered from the atmosphere for the growth of vegetable oil crops. The process of utilizing biodiesel in the IC engines for transport as well as other applications, is gaining momentum recently. The European Commission proposed a 12percent market share for bio-fuels by the year 2020. In Austria, biodiesel from rapeseed oil is commercialized. Tax benefits in Austria and Germany encourage the use of 100 percent biodiesel fuel in ecological sensitive areas [6]. The use of bio-fuels is predominantly led by legislation, but it is also supported by incentives such as reduced tax and agricultural subsidies on bio-fuel feedstock crops. In Europe the EU Transport Policy is targeting replacement of 20% of conventional on-road fuels with substitute fuels by 2020 [11]

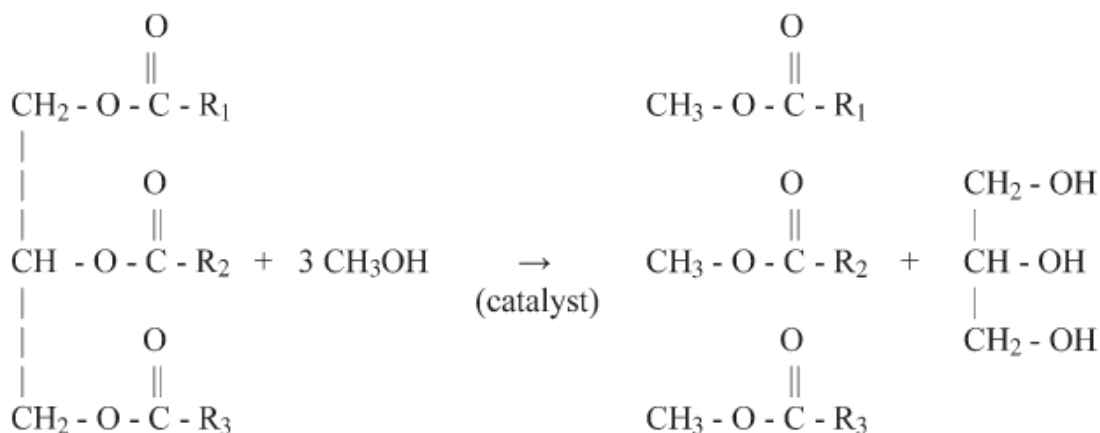
## 2. BIODIESEL FUEL

"Biodiesel is defined as mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats" which conform to American Society of Testing Materials (ASTM D5453) [12].

Biodiesel is an alternative fuel for diesel engines that is produced by chemically reacting a vegetable oil or animal fat with an alcohol such as methanol. The reaction requires a catalyst, usually a strong base, such as sodium or potassium hydroxide, and produces new chemical compounds called methyl esters. It is these esters that have come to be known as biodiesel [13]. Different ways have been considered to reduce the high viscosity of vegetable oils. But the transesterification seems to be the best choice. Although there are many ways and procedures to convert vegetable oil into a diesel-like fuel, transesterification process was found to be the most viable oil modification process. At present, the most common way to produce biodiesel (BD) is to trans esterify triacylglycerols in vegetable oil or animal fats with an alcohol in the presence of an alkali or acid catalyst [14].

Biodiesel is produced through a process known as transesterification, as shown in the equation below. Where,  $R_1$ ,  $R_2$ , and  $R_3$  are long hydrocarbon chains. It's sometimes called fatty acid chains. Although the research did not anticipate the production of alkyl esters for fuel, most of the processes for biodiesel production were developed in the early 1940s. Researcher received a patent for a process that added about 1.6 times the theoretical amount of an alcohol, such as methanol, which contained 0.1 to 0.5%

sodium or potassium hydroxide, to an oil or fat. When performed at 80 °C, this process provided 98% conversion to alkyl esters and high-quality glycerol [14].



**Triglyceride**
**Methanol**
**Mixture of fatty esters**
**Glycerin**

Biodiesel, derived from vegetable oils, is the most promising alternative fuel to diesel due to the following reasons [15, 16].

- Biodiesel can be used in the existing engine without any modifications.
- Biodiesel is made entirely from vegetable sources; it does not contain any sulphur, aromatic hydrocarbons, metals or crude oil residues.
- Biodiesel is an oxygenated fuel; emissions of carbon monoxide and soot tend to reduce.
- Unlike fossil fuels, the use of Biodiesel does not contribute to global warming as CO<sub>2</sub> emitted is once again absorbed by the plants grown for vegetable oil/biodiesel production. Thus, CO<sub>2</sub> balance is maintained.
- The Occupational Safety and Health Administration classify biodiesel as a non-flammable liquid.
- The use of biodiesel can extend the life of diesel engines because it is more lubricating than petroleum diesel fuel.
- Biodiesel is produced from renewable vegetable oils/animal fats and hence improves the fuel or energy security and economy independence.

### 3. PRESENT WORK OBJECTIVES

The desirability of developing biodiesel from different tree born oil seeds and decreasing the dependency on petroleum-based fuels has been discussed by many over the last few decades. However, some of the important issues of biodiesel like resistant to oxidative degradation during storage, changes in important fuel properties like FFA, viscosity etc. which are increasingly important issues for the successful development and viability of alternative fuels are not analysed to a substantial extent. Oxidation of unsaturated esters in biodiesel occurs by contact with air and other peroxidising conditions during long term storage. Thus, oxidative stability is an important issue that biodiesel research must address since oxidation product may impair fuel quality and, subsequently, engine performance. Present work objectives are to address these important issues long term acceptability of this new member called biodiesel in the existing family of diesel engines. The work comprises of storing the non-edible oils of *Jatropha* and *Pongamia* for long term (24 months) and then study changes in storage sensitive properties of oils like FFA, Acid Value, Viscosity etc. The present work also comprises of studying the effect of

storage oil on FFA and yield of biodiesel. The present work also discusses the impact of storage of oil and biodiesel on storage container of biodiesel. This work also focuses on effect of biodiesel obtained from stored oils on engine performance.

#### **4. PRESENT WORK OBJECTIVES**

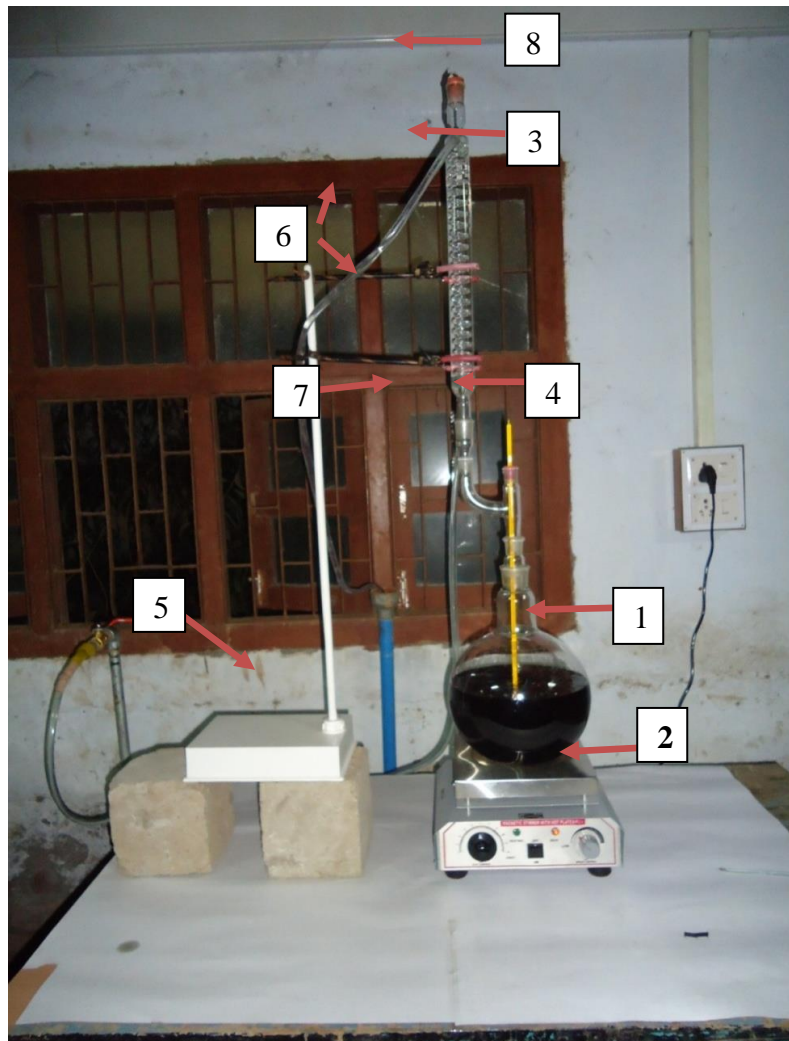
The desirability of developing biodiesel from different tree born oil seeds and decreasing the dependency on petroleum-based fuels has been discussed by many over the last few decades. However, some of the important issues of biodiesel like resistant to oxidative degradation during storage, changes in important fuel properties like FFA, viscosity etc. which are increasingly important issues for the successful development and viability of alternative fuels are not analysed to a substantial extent. Oxidation of unsaturated esters in biodiesel occurs by contact with air and other peroxidising conditions during long term storage. Thus, oxidative stability is an important issue that biodiesel research must address since oxidation product may impair fuel quality and, subsequently, engine performance. Present work objectives are to address these important issues long term acceptability of this new member called biodiesel in the existing family of diesel engines. The work comprises of storing the non-edible oils of *Jatropha* and *Pongamia* for long term (24 months) and then study changes in storage sensitive properties of oils like FFA, Acid Value, Viscosity etc. The present work also comprises of studying the effect of storage oil on FFA and yield of biodiesel. The present work also discusses the impact of storage of oil and biodiesel on storage container of biodiesel. This work also focuses on effect of biodiesel obtained from stored oils on engine performance.

#### **5. EXPERIMENTAL SET UP**

##### **4.1 TRANSESTERIFICATION PROCESS**

The most common way to produce biodiesel is by transesterification. In reaction, triglycerides, as the main components of vegetable oils, react with an alcohol to produce fatty acid mono alkyl esters and glycerol. Methanol is the most common alcohol because of its low price compared to other alcohols. In this case, the reaction is referred to as metanalysis. The stoichiometry of methanol reaction requires 3 mol of methanol and 1 mol of triglyceride to give 3 mol of fatty acid methyl ester and 1 mol of glycerol. This reaction, in turn, consists of three consecutive reversible reactions with immediately formation of di-glycerides and mono-glycerides. After the reaction, the glycerol is separated by settling or centrifuging and the layer obtained as purified to be used in its traditional applications (the pharmaceutical, cosmetic and food industries) or in its recently developed applications (animal feed, carbon feedstock in fermentations, polymers, surfactants, intermediates and lubricants). The biodiesel phase is also purified before being used as diesel fuel in order to fulfil the EN 14214 standards. One of the advantages of this fuel is that the raw materials used to produce it are natural and renewable. All these types of oils come from vegetables or animal fat (refine, crude or frying oils and fats) making it biodegradable and nontoxic. There are different types of catalyst: basic, acid, ion exchange resin, enzymes and supercritical fluids. However, the basic catalyst is the most commonly used in industry, because the process proves faster and the reaction conditions are moderate.

#### 4.1.1. TRANSESTERIFICATION PROCESS SETUP



**Figure: 4.1 Transesterification process setup**

- |                                   |                       |
|-----------------------------------|-----------------------|
| [1]. Glass Reactor                | [5] Retard Stand      |
| [2] Electrical Heater and Stirrer | [6] Clamps            |
| [3] Glass condenser               | [7] Cooling Water In  |
| [4] Glass Thermometer             | [8] Cooling Water Out |

Biodiesel was prepared from raw non edible oil of Pongamia and Jatropha for the present study. Following process was executed for transesterification. The transesterification process was followed for biodiesel preparation.

For making of the Pongamia and Jatropha biodiesel the catalyst used was KOH and mixed with methanol for separating ester and glycerol. For the quantity of 1 kg of vegetable oil the proportion of catalyst KOH is was 15gm and the proportion of methanol was 250gm. This proportion is mixed in to a glass reactor and stirred. Figure 4.1 shows the transesterification set up which was designed and developed in house at IC Engines Lab. The set-up work comprised of 05 liter capacity biodiesel reactor for producing biodiesel using the process of transesterification. The reaction was carried out at a temperature of 600C for 3 hr. The raw oil will be stirred and heated using the electric heater and stirrer. Since the raw oil is to be mixed with methyl alcohol during the process of transesterification care was taken during heating such that the methyl alcohol should not vent away during the heating process. This

is mainly because methyl alcohol will evaporate after 65<sup>0</sup>C. To ensure this the propose set up consist of tube in tube type condenser through which water flows continuously. Thermometer was incorporated into the system to ensure that the temperature in the range of 60-65<sup>0</sup>C was maintained throughout the process. Subsequent process like separation and washing process is follow to get biodiesel.

Biodiesel was prepared from raw non edible oil seeds of Pongamia by transesterification. The biodiesel yield was estimated after the reaction, collection from the upper layer of the decanter and extra methanol removal. The produced biodiesel weight relative to the initial used vegetable oil was taken as the biodiesel yield. Theoretically, if 100 kg of triglycerides react with 10 kg of methanol, theoretical results should be 100 kg of biodiesel and 10 kg of glycerol. After transesterification process FFA value and Yield of different biodiesels are shown in table 4.1.

**Table 4.1: FFA value% Vs %Yield of Oil**

SAMPLE	FFA Value (%)	%Yield
PONGAMIA NEW OIL	3.78	82.50
PONGAMIA OLD OIL	5.75	80

Thus, from above it can be concluded that the FFA value increases with the storage of oil. The increase in the FFA has resulted in net decrease in the yield of biodiesel.

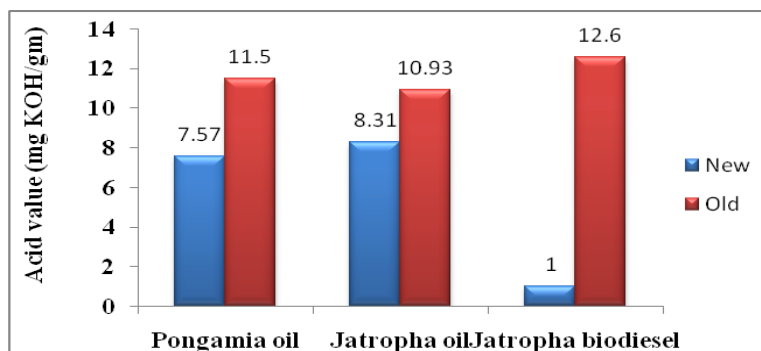
## 4.2 MONITORING BIODIESEL QUALITY DURING STORAGE

Some of the basic properties of biodiesel like viscosity, calorific value, acid value, peroxide value etc. were determined for the new and old (24 months) samples with the in-house available facility. Generally, changes in Acid value, Peroxide value and Viscosity are treated as measure for analyzing the quality of oil and biodiesel in long term storage.

### 4.2.1 ACID VALUE

**Table 4.2.1: Acid value of samples**

Sr. No.	Sample	Acid Value (mg KOH/gm)
1	Pongamia new oil	7.57
2	Pongamia old oil	11.5
3	Jatropha new oil	8.314
4	Jatropha old oil	10.93
5	Jatropha new biodiesel	1.0
6	Jatropha old biodiesel	12.06



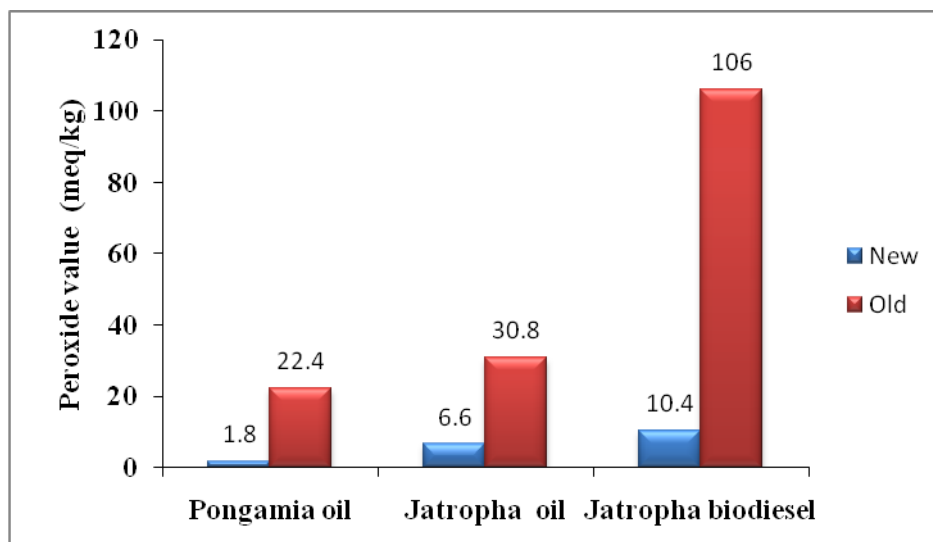
**Fig 4.2.1: Evaluation of the acid value of new and old samples**

From figure 4.1 it is clear that due to storage (24 months) there exist the increase in the FFA values of oils and biodiesel. The increase in the acid value of stored biodiesel is much higher than that of vegetable oils. In case of biodiesel the acid value increases with increase in storage due to the hydrolysis of fatty acid methyl ester to fatty acids. The increase in the acid value of Pongamia oil with storage was observed of the order of 51 % where as in case of Jatropha oil the increase in acid value with storage was observed of the order of 31%. Thus, storage influence was observed to be more in case of Pongamia oil as compared to that of Jatropha oil.

#### 4.2.2 PEROXIDE VALUE

**Table 4.2.2: Peroxide value of samples**

Sr. No.	Sample	Peroxide Value (meq/kg)
1	Pongamia new oil	1.8
2	Pongamia old oil	22.4
3	Jatropha new oil	6.6
4	Jatropha old oil	30.8
5	Jatropha new biodiesel	10.4
6	Jatropha old biodiesel	106



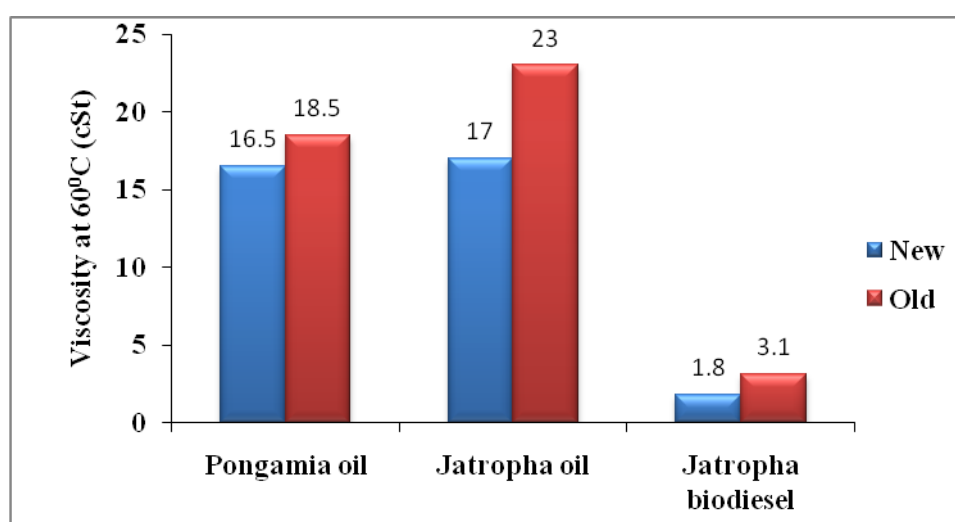
**Fig 4.2.2: Evaluation of the peroxide value of new and old samples**

In agreement with the results of the determination of the acid value, the peroxide value increases with storage time. From figure 4.2.2 it is clear that the peroxide value of stored oils and biodiesel increases with storage time. In case of Pongamia oil the increase in peroxide value with the storage time was almost 12 times where as in case of Jatropha it was almost 4.5 times. Thus, higher increase in peroxide value for Pongamia oil was observed as compared to that of Jatropha oil indicating poor storage stability as compared to Jatropha. Peroxide values were measured in milliequivalents of peroxide per kg of sample. Similarly in case of biodiesel the increase in peroxide value with storage was almost 10 times.

### 4.2.3 VISCOSITY

**Table 4.2.3: Viscosity of samples**

Sr. No.	Sample	Viscosity at 60 <sup>0</sup> C (cSt)
1	Pongamia new oil	16.5
2	Pongamia old oil	23
3	Jatropha new oil	16.5
4	Jatropha old oil	18.2
5	Jatropha new biodiesel	1.8
6	Jatropha old biodiesel	3.1



**Fig 4.2.3: Evaluation of the viscosity of new and old samples**

Viscosity has been found to increase with chain length (number of carbon atoms and with increasing degree of saturation). FFAs are responsible for higher viscosity than the corresponding methyl or ethyl ester. It is also reported that oxidation of methyl ester begins with build-up of peroxide, viscosity starts to increase only after the peroxides have reached a certain level. During storage the viscosity of methyl esters increases by the formation of

more polar oxygen containing molecules and also by the formation of oxidized polymeric compounds that can lead to formation of gums and sediments that clog filters. In this work vegetable oils and biodiesel shown increase in viscosity after long term storage. From figure 4.2.3 it is clear that the increase in viscosity of Pongamia oil was of the order of 12 % whereas in case of Jatropha oil it was of the order of 35 %. In case of biodiesel of Jatropha, the increase in viscosity was very steep and was of the order of 72 %.

### 4.3 Engine performance

The investigations on storage stability of fuel particularly biodiesel concern the property changes occurring during storage and their influence on engine performance. The fuel property changes of biodiesel obtained from new and old Pongamia oil and their effect on combustion, performance and emission characteristics are presented and discussed here. The properties of new and old fuel samples are measured according to their ASTM standards and provided in table.



4.3.1 Fuel properties

**Table 4.3.1: Pongamia biodiesel properties derived from new oil (A-PME) and old oil (B-PME)**

Sr. No.	Properties	Unit	A-PME	B-PME
1	Kinematic viscosity at 60 <sup>0</sup> C	cSt	2.28	2.50
2	Acid value	mg KOH/gm	0.84	0.84
3	Density	kg/m <sup>3</sup>	885	890
4	Calorific value	kcal/kg	10370	10390

**TABEL 4.3.2: Observation table for engine test for Pongamia biodiesel derived from new oil (a-pme)**

sr. no	Load (kg)	Time (sec) for fuel consumption 10cc	Rpm	Exhaust gas temperature	Water outlet temperature	Time mass flow rate water
1	0	94	1540	140	38.9	35
2	4	73	1520	164	40.4	34.7
3	8	58	1500	198	42.2	33.6
4	12	46	1480	241	43.7	33.5
5	16	49	1460	287	45.8	32.3

**TABLE 4.3.3 Results**

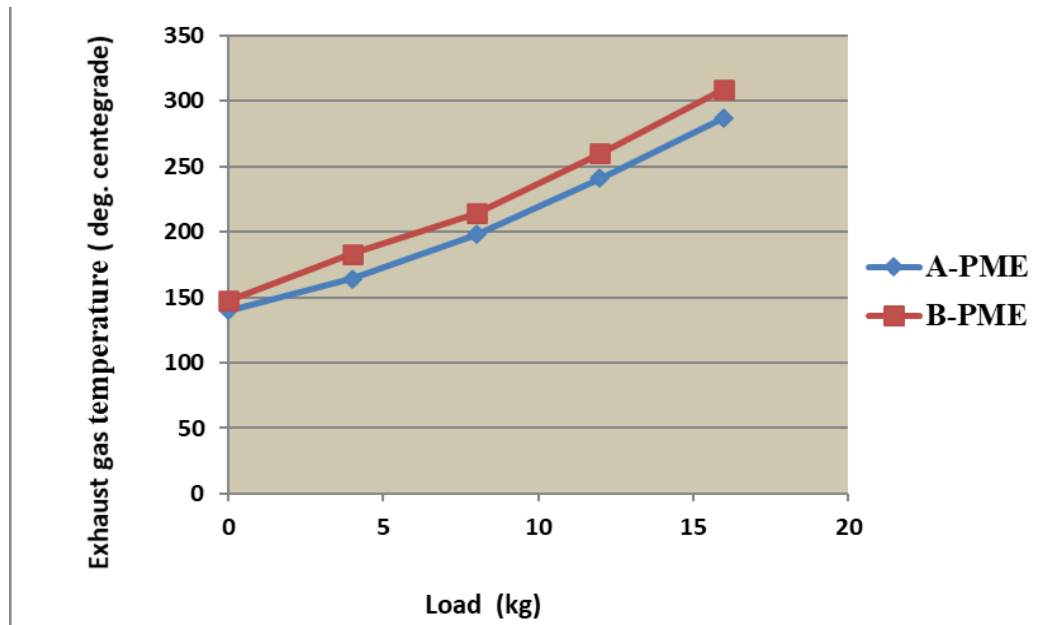
Load (kg)	Time (sec)	Mass Flow Rate mf	CV	mf*CV	BP (W)	BP/ mf*CV	Efficiency %
0	94	0.0942	43346.6	4081.69	0	0	0
4	73	0.1212	43346.6	5255.03	687.05	0.1307	13.07
8	58	0.1526	43346.6	6614.7	1356	0.2050	20.5
12	46	0.1924	43346.6	8339.5	2006.93	0.2407	24.07
16	39	0.2269	43346.6	9836.34	2639.75	0.2684	26.84

**TABEL4.3.4 Observation table for engine test for Pongamia biodiesel derived from old oil (b-pme)**

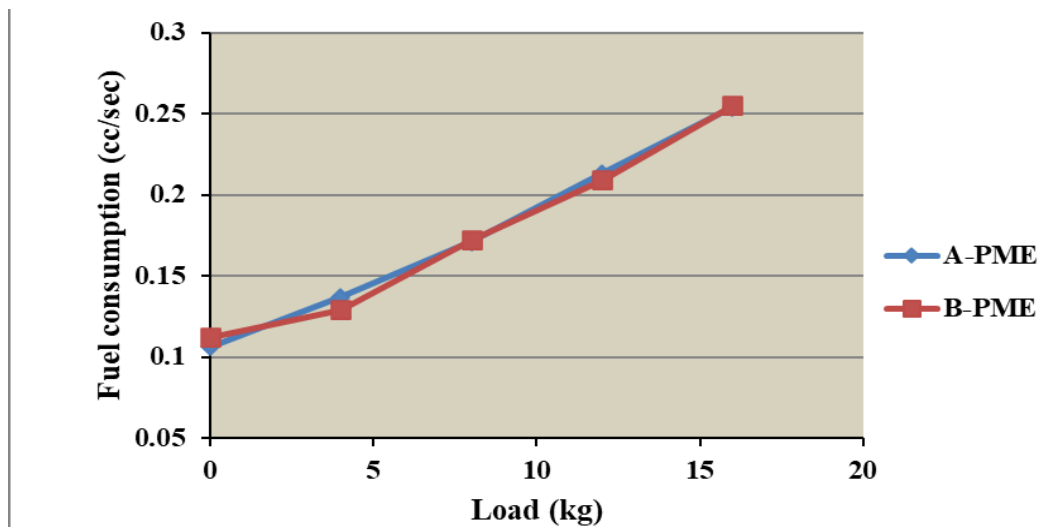
Sr.No	Load (kg)	Time (sec) for fuel consumption 10cc	Rpm	Exhaust Gas Temperature (° C)	Water Outlet Temperature	Time (sec) for mass flow rate water
1	0	89	1540	147.2	377	32
2	4	77	1520	183	39.	33.8
3	8	59	1500	214	41.5	33.3
4	12	47.66	1490	260	43.5	33.1

**TABLE 4.3.5 RESULTS**

Load (kg)	Time (sec)	Mass Flow Rate m <sub>f</sub>	CV	m <sub>f</sub> *CV	BP (W)	BP/m <sub>f</sub> *CV	Efficiency %
0	89	0.1	43430.2	4343.02	0	0	<b>0</b>
4	77	0.1156	43430.2	5020.5	687.05	0.1369	<b>13.69</b>
8	58	0.1534	43430.2	6664.29	1356.07	0.2035	<b>20.35</b>
12	47	0.1894	43430.2	8224.01	2020.5	0.2457	<b>24.57</b>
16	39	0.2282	43430.2	9910.99	2639.75	0.2663	<b>26.63</b>



**Figure 4.3.1 Comparison of exhaust gas temperature of new oil and old oil derived biodiesel fuels at different load condition**



**Fig 4.3.2 Comparison of fuel consumption of new oil and old oil derived biodiesel fuels at different load condition**

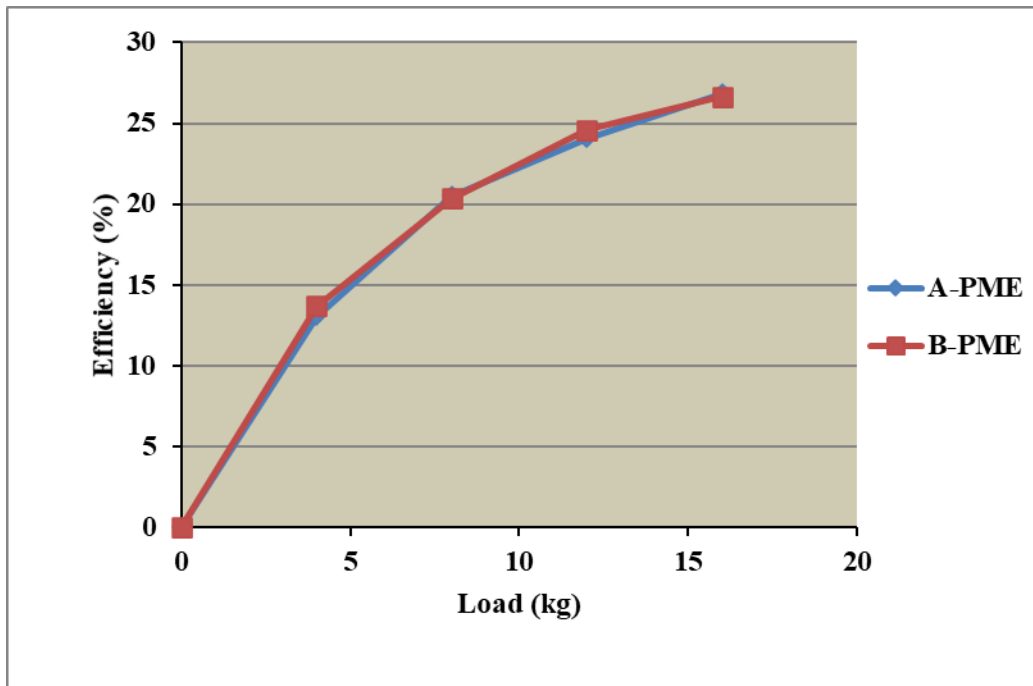


Figure 4.3.3 Comparison of fuel consumption of new oil and old oil derived biodiesel fuels at different load condition

#### 4.4 MATERIAL COMPATIBILITY OF STORAGE CONTAINERS

In order to understand the compatibility of storage material of oil, the storage containers were observed for a period of two years and changes in shape of the container i.e., deformation of containers/leakages were observed during this period.

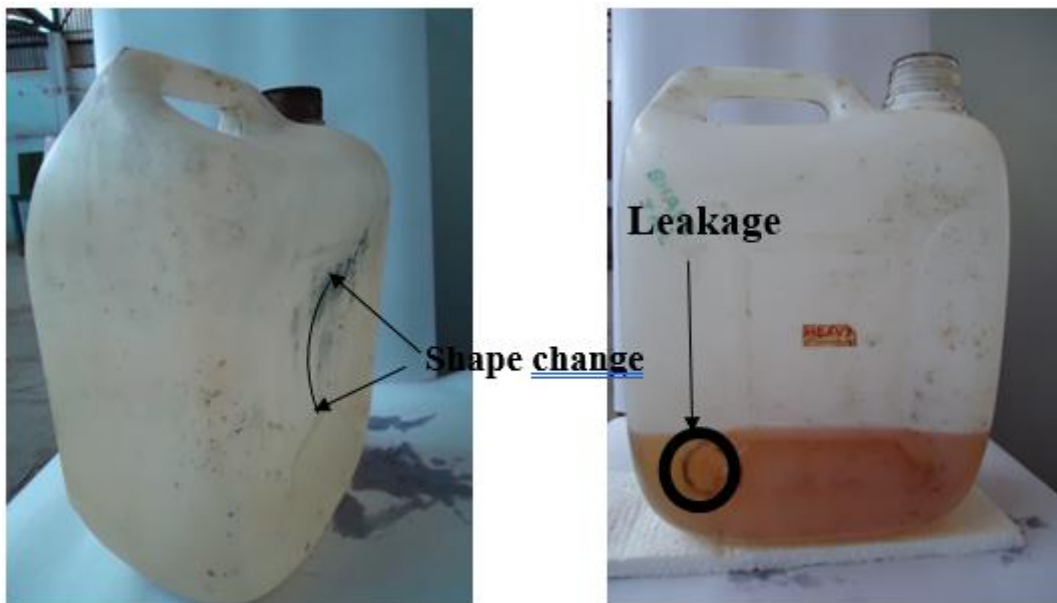


Figure 4.4: Storage issues - Plastic Material Compatibility

Fig 4.4 shows changes in shape of the container as well as formation of leakage when stored for long period. It is suggested that storage of oils should be in stainless steel container rather than plastic container when it is to store for a long period.

## CONCLUSION

In order to substitute the diesel fuel with alternative fuel like non-edible vegetable oil and biodiesel in the existing engine, an attempt was made to address the important issue of storage stability of biodiesel. Jatropha and Pongamia oils were selected for the present study and were stored for 24 months. Changes in certain properties which are important from storage point of view were monitored for the storage period. Effect of biodiesel obtained from new and old biodiesel on engine performance was also studied. Following conclusions can be drawn from the present work. FFA value of oils (which directly influences the biodiesel yield) increases with long term storage of oils.

The increase in FFA values of vegetable oils ultimately results in increase in the acid value of oils. The increase in the acid value of Pongamia oil with storage was observed of the order of 51 % where as in case of Jatropha oil the increase in acid value with storage was observed of the order of 31%. Thus, storage influence was observed to be more in case of Pongamia oil as compared to that of Jatropha oil.

The increase in acid value of stored biodiesel was much higher than that of vegetable oils. This was mainly due to the hydrolysis of fatty acid methyl ester to fatty acids. It can also be concluded that the peroxide value of stored oils and biodiesel increases with storage time. In case of Pongamia oil the increase in peroxide value with the storage time was almost 12 times where as in case of Jatropha it was almost 4.5 times. Thus, higher increase in peroxide value for Pongamia oil was observed as compared to that of Jatropha oil indicating poor storage stability as compared to Jatropha. Similarly in case of biodiesel the increase in peroxide value with storage was almost 10 times.

Viscosity has been found to increase with chain length (number of carbon atoms and with increasing degree of saturation). FFAs are responsible for higher viscosity than the corresponding methyl or ethyl ester.

It can also be concluded that the increase in viscosity of Pongamia oil was of the order of 12 % whereas in case of Jatropha oil it was of the order of 35 %. In case of biodiesel of Jatropha, the increase in viscosity was very steep and was of the order of 72 %. Engine performance test on biodiesel obtained from old and new Pongamia oil revealed that there was no significant difference observed in overall engine performance.

With reference to selection of plastic container for storage point of view, it can be stated that with long term storage of non-edible vegetable oil, leakages and deformation occurs. It is suggested that for long term storage of oil it is always better to use stainless steel tanks. Thus, in general it can be concluded that biodiesel can very well act as a substitute with diesel for CI engines. However certain improvements in the storage conditions such as storage in stainless steel container with suitable anti-oxidants will ensure the long-term storage stability of vegetable oils and biodiesel fuel.

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