MATERIALS AND METHODS

Non-edible oils are being successfully utilized for the production of biodiesel. Rubber seed oil being a by product can have economic advantages in its use. At present, rubber seed oil finds industrial application in very limited fields (Joseph, 2004; Ramadhas et al., 2006 and Abdullah and Salimon, 2009). In order to consider it as one of the source for biodiesel a detailed investigation was carried out.

1. Collection of rubber seeds

Rubber seeds required for the extraction of oil for the present study were collected from the rubber tree plantations at Kanyakumari District of Tamil Nadu in India. Seeds were collected (once a year) for a period of two years (2006 - 2008). The collected seeds were transported to the laboratory and sun dried for one week. It was then cleaned and weighed after removing the hard seed coat.

2. Extraction of oil

Extraction of oil from the kernel for the study was carried out by two modes. In the first mode, the extraction of oil was made through a commercial oil expeller. In the second mode, the extraction was made using solvent extraction method. Solvent extraction method was carried out as per the procedure described in AACC, 1991 method. All determinations were repeated thrice.

3. Moisture content of the rubber seed oil

Moisture content of the oil was determined by drying a known weight of the sample to a constant weight in an electric oven at 103 ± 2 °C.
A weighing crucible was placed in an electric oven at 103 ± 2 °C for about 17 h (ISTA, 2005). The crucible was removed, cooled in a desiccator and its weight was recorded (A). About 5 g of the oil was transferred to the crucible and then weighed (B). It was kept in the oven at 103 ± 2 °C for 1 h. Then the crucible was removed from the oven. It was cooled in the desiccator and weighed (C).

The process was repeated until the concordant weight was obtained. The moisture content was calculated as follows:

\[
\text{Moisture content (\%) } = \frac{B \times C}{B \times A} \times 100
\]

Where,

A = Initial weight of the crucible (g).
B = Initial weight of the sample + crucible (g).
C = Final weight of the sample + crucible (g).

4. Physico-chemical characteristics of rubber seed oil

4.1 Acid value

Acid value of the samples was assessed as per the procedure described in ASTM D 664 – 07. Free fatty acid was determined from the acid value.

4.2 Iodine value

Iodine value of the samples was estimated as per the procedure followed by ASTM D 1959 – 97.

4.3 Saponification value

Saponification value of the samples was assessed as per the procedure described in ASTM D 94 – 07.
4.4 Peroxide value

Peroxide value test was carried out using the methods of AOAC (1990).

4.5 Specific gravity

ASTM D 1250 – 08 methods was used to determine the specific gravity of the samples.

4.6 Kinematic Viscosity

Kinematic viscosity of the samples was determined using a Redwood viscometer (Deep vision instrument) according to the procedure followed by Sangha et al. (2004).

Water was filled in the bath and was heated to few degrees above testing temperature. The test sample was filtered and poured into the oil cup of viscometer. A 50 ml measuring cylinder was placed at few cm below the jet of viscometer. The bath valve in the oil cup of viscometer was lifted to allow the oil to drop down. The time taken by the oil to fill 50 ml sample in the measuring cylinder was noted by lifting the bath valve in oil cup of viscometer. The experiment was repeated and the mean values were taken.

The kinematic viscosity of the sample was calculated by using the following equations. If the time taken for the sample to reach the 50 ml graduation mark of the measuring cylinder was within 20 to 80 seconds then the viscosity was calculated from the equation:

\[ \eta = 0.00264 \left( \frac{t}{20} \right) - \frac{1.9}{t} \quad \text{(where } t = 20 \text{ to } 80 \text{ sec)} \]
If the time taken for the sample to reach the 50 ml graduation mark of the measuring cylinder ranged from between 80 to 2000 seconds, the viscosity was calculated from the equation:

\[ \eta = 0.00247 \times (t) - 0.65 / t \]  

(4.7) Calorific value

The calorific value of the samples was assessed according to the procedure described in ASTM D 240 - 09.

4.8 Elemental analysis

Carbon, hydrogen, nitrogen and sulphur of the sample were analysed using the elemental analyser (Elementar vario EL III – Germany).

5. Transesterification process

In order to achieve high efficiency of biodiesel production from rubber seed oil, this study was taken up with two types of catalyst namely homogeneous and heterogeneous catalyst. If the reactants and catalysts are in same phase, when mixed it is said to be a homogeneous catalyst. Similarly, if the reactants and catalysts are in different phase when mixed it is said to be heterogeneous catalyst. The main advantage of using heterogeneous catalyst is that it can be reused for several times. Therefore, an attempt has been made on transesterifying the oil using homogeneous and heterogeneous catalyst in lab scale method and reactor (pilot plant) method.

5. a. Lab scale method

Homogeneous catalysts

Acid esterification process

One litre of filtered raw oil was heated in a 2 litres conical flask to 100 °C for 5 min to expel the moisture content. Performance of acids (hydrochloric acid, sulphuric
acid and orthophosphoric acid) as catalysts at the rate of 2, 3, 4, 5, 6 and 7 ml/l of oil with methanol to oil molar ratio of 9:1 was analysed in favour of reducing the free fatty acid present in the oil.

The acid mixed in methanol was added to the oil and heated by keeping it over a magnetic stirrer (2MLH Remi stirrer) at a speed of 600 rpm. It was then transferred to a separating funnel and waited for separation. After 5 h, excess methanol, acid and water mixture rose to the top and the acid esterified oil settled at the bottom of the funnel. The acid which caused the maximum reduction of free fatty acid in oil at its low quantity of application was selected for the present study. If the free fatty acid content did not reduce to 3 percent and below, it was treated once again. The second treatment was not observed to be advantageous. The above trials were carried out at different temperatures (40, 45, 50, 55 and 60 °C) so as to know the optimum temperature required for the reaction. Similarly, the effect of reaction time was also investigated (30, 45, 60 and 75 minutes) so as to optimize the time required for the completion of reaction.

The FFA reduction was calculated as follows:

\[
\text{FFA reduction (\%) = } \frac{\text{FFA in oil} - \text{FFA reduction after esterifying the oil}}{\text{FFA in oil}} \times 100
\]

Alkaline transesterification

The acid esterified oil was then taken in a 2 litres conical flask and the same was heated to 55 °C. To investigate the effect of different alkaline catalysts on transesterification process, three types of commonly used conventional homogeneous base catalysts (i.e) sodium hydroxide, potassium hydroxide and sodium methoxide were tried where methanol was the alcohol. All the catalysts used were of laboratory grade. The performance of each of these catalysts, on the acid esterified oil was evaluated at a doses of 2, 3, 4, 5 and 6 g/l of oil under different
molar ratio of alcohol to oil 6:1 at various reaction temperatures (40, 45, 50, 55 and 60 °C) and duration of 30, 45, 60 and 75 minutes. After the reaction was completed the mixture was transferred to a separating funnel. The set up was kept undisturbed for 8 h for separation. Glycerol being the by product moved to the lower part and the methyl ester moved to the upper part of the funnel. Glycerol was then decanted. The product (methyl ester) was washed with 600 ml of distilled water for 3-4 times till the unreacted alkali was removed (indicated by litmus paper). Finally, it was heated to 110 °C to eliminate the water content. The yield of the methyl ester was estimated according to Rashid and Anwar (2008).

\[
\text{Yield of methyl ester (\%)} = \frac{\text{Methyl ester produced (g)}}{\text{Oil taken for the reaction (g)}} \times 100
\]

**Heterogeneous catalyst**

**Acid esterification**

Perusal of the literature (Wang et al., 2006; Liu et al., 2008; Sharma et al., 2008 and Omar et al., 2009) indicated that heterogeneous catalysts offered better catalytic activity than homogeneous catalysts. Therefore, in the present study, ferric sulphate \([\text{Fe}_2(\text{SO}_4)_3]\) was tried as a heterogeneous acid catalyst and calcium oxide \([\text{CaO}]\) was employed as a heterogeneous base catalyst.

One litre of rubber seed oil was mixed with 9:1 molar ratio of methanol to oil at various rates (1, 1.5, 2, 2.5, 3 and 3.5 g/l) of ferric sulphate in a 2 litres conical flask and boiled for a specified period of time and temperature. The mixture was left overnight for separation. Two layers were formed. The upper layer containing the acid esterified oil was then subjected to the second step transesterification and the bottom layer contained water with ferric sulphate.
The FFA reduction was calculated as follows:

\[
\text{FFA reduction (\%) = } \frac{\text{FFA in oil} - \text{FFA reduction after esterifying the oil}}{\text{FFA in oil}} \times 100
\]

The level of FFA in acid esterified oil was calculated as

\[
\text{FFA level (\%) = } \frac{100 - \text{FFA reduction} \times \text{FFA in oil}}{100}
\]

Recovery of the catalyst

The bottom layer containing ferric sulphate after the methanol recovery of each performance was drained in a crucible. The recovered ferric sulphate was reclaimed by ashing as per the method of Wang et al. (2006). It was then ashed at 460 °C for 5 h in a muffle furnace so as to remove the organic impurities. The recovered ferric sulphate was collected for reuse. The recovery ratio of the catalyst was calculated by the amount of recovered one over the fresh one.

Alkaline transesterification

Transesterification reactions were carried out in a 2 litre conical flask. First, the catalyst calcium oxide (at a rate of 1, 1.5, 2, 2.5, 3 and 3.5 g) was dispersed in methanol to oil molar ratio of 6 : 1 under magnetic stirring at a speed of 600 rpm. Then, the acid esterified oil was added into the mixture and heated. Different durations and temperatures were tried. It was then transferred to a separating funnel and kept undisturbed for separation. Three phases were observed. The upper layer was biodiesel, the middle layer was glycerol and the lower layer was a mixture of solid calcium oxide. The solid calcium oxide was drained out first for reuse. It should be noted that, although water washing step is not necessary for calcium oxide catalysed reaction but for the comparison it was followed.
The upper phase (methyl ester) was washed with distilled water of 600 ml for 3 - 4 times. Finally, it was heated to 110 °C to eliminate the water.

\[
\text{Yield of methyl ester (\%) } = \frac{\text{Methyl ester produced (g)}}{\text{Oil taken for the reaction (g)}} \times 100
\]

**Water wash**

The purpose of water wash was to remove the unreacted alcohol, catalyst or glycerine present in the biodiesel. Unreacted alcohol decreases the flash point of biodiesel. Biodiesel with 0.2 percent alcohol does not found to meet ASTM fuel standards. Therefore, it was necessary to remove the unreacted alcohol from biodiesel. The excess methanol in biodiesel corrodes the fuel injection system and hence it should be separated from the biodiesel. Water wash would remove any soap and unreacted alkali if present in the biodiesel.

**Drying biodiesel**

Drying was ascertained by heating the washed fuel to 110 °C in an open container until there was no more steam in the fuel. The resultant fuel should be clear and amber coloured liquid. This heating process would drive off any traces of remaining alcohol. If the fuel appeared cloudy after drying, then drying cycle should be repeated and it might be due to the presence of non – water soluble contaminants in the fuel (such as mono and di glycerides).

**Testing of biodiesel**

After water washing and drying of the biodiesel, the completion of the reaction was confirmed by the following tests.

1. 25 ml of biodiesel was fully dissolved in 225 ml of methanol forming a clear bright phase. If any undissolved material were observed at the bottom, it could be inferred that the reaction does not proceeded to completion.
2. Emulsification

Biodiesel was mixed with water (50 / 50 mix) and shaken vigorously. If the resulting mixture separated quickly and the biodiesel phase on the top appears clear and bright and the water phase at the bottom appears clear and free of debris, then the biodiesel produced is clean.

In case, if the resulting mixture does not settle out within a few minutes, then the fuel still contains excess soap. Care must be taken during washing that it should not be agitated vigorously.

5. b. Pilot plant method

Production of methyl ester was tested in a 10 litres capacity reactor developed in our laboratory (Baskar, 2006). The reactor had a single walled cylindrical shell. Circular disc with rubber gasket have been provided on the top of the reactor in order to avoid any leakage from the system. A funnel with a gate valve was fitted on the top of the reactor for feeding the reactants. The reactor had a motor driven stirrer, a built in heater and a sensor. The stirring and heating processes were controlled externally by an electronic device.

The sensor indicates the reaction temperature of the content. Condensation unit present at the top of the cylinder helps to distill out methanol and water. Inlet and outlet arrangements provided at the bottom and top of the reactor facilitated the entry and exit of the water wash. The separation of the layer could be seen through a window fitted with a glass plate on the side wall of the reactor. A gate valve at the bottom helped to drain out the glycerol and the ester.

Fig.4 Pilot scale plant for biodiesel production (Baskar, 2006)
The trials in the reactor were carried out as per the procedure being followed in the lab scale methods (5.a.).

6. Quantitative analysis of methyl ester

The quantitative analysis of methyl ester were analysed using Fourier Transform Infra Red (Perkin-Elmer-Paragon IR spectrophotometer).

7. Properties of biodiesel

Physico-chemical characteristics such as free fatty acids, acid value, iodine value, saponification value, peroxide value, specific gravity, viscosity, calorific value and elemental analysis of biodiesel thus produced from the most excellent homogeneous alkaline catalysts. They were analysed according to the procedure followed in the section 4.1 – 4.8 of the Materials and Methods. Following properties were also analysed.

**pH**

pH of the methyl ester was analysed using a pH meter (Elico India 101 E) immediately after its production.

**Cetane number (CN)**

Cetane number of the methyl ester was calculated based on the equation of Azam *et al.*, 2005.

\[
\text{Cetane number (CN)} = 46.3 + \frac{5458}{\text{SN}} - 0.225 \times \text{IV}.
\]

Where,

\[
\text{SN} = \text{Saponification number of the methyl ester.}
\]

\[
\text{IV} = \text{Iodine value of the methyl ester.}
\]
Copper corrosion

The copper strip corrosion test was performed as per the procedure contained in ASTM D 130 - 04. 30 ml of biodiesel was taken in a test tube of 150 ml capacity and kept maintained at 50 ± °C using a water bath. A copper strip was slid into it and kept for 3 hours. The copper strip was then removed, washed in iso octane and allowed to dry. It was then subjected to copper strip corrosion standards. Freshly polished strip was the control.

<table>
<thead>
<tr>
<th>Classification</th>
<th>Designation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Freshly polished strip (control)</td>
<td>----</td>
<td>B</td>
</tr>
<tr>
<td>1 Slight tarnish</td>
<td></td>
<td>a. Light orange, almost the same as freshly polished strip.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>b. Dark orange.</td>
</tr>
<tr>
<td>2 Moderate tarnish</td>
<td></td>
<td>a. Claret red</td>
</tr>
<tr>
<td></td>
<td></td>
<td>b. Lavender.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>c. Multicoloured with lavender blue or silver or both, overlaid on claret red.</td>
</tr>
<tr>
<td>3 Dark tarnish</td>
<td></td>
<td>a. Magenta overcast on brassy strip.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>b. Multicoloured with red and green showing (Peacock) but no gray.</td>
</tr>
<tr>
<td>4 Corrosion</td>
<td></td>
<td>a. Transparent black, dark grey or brown with peacock green barely showing.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>b. Graphite or lusterless black.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>c. Glossy or jet black.</td>
</tr>
</tbody>
</table>

Conradson carbon residue

The carbon residue test was conducted according to the procedure in ASTM D 189 - 06. 10 gram of the sample was weighed in a porcelain crucible and placed in
a skidmore crucible. The skidmore crucible was then kept in an iron crucible and it was loosely covered. The iron crucible was subjected to high strong flame from gas burner. The pre-ignition period was 10 min. Then the gas burner was tilted after the appearance of smoke and the flame intensity was reduced when the ignited vapours burn uniformly. The burning time for the vapour was 13 min. The iron crucible was heated to cherry red hot and was maintained for 7 min until no blue smoke emits from the apparatus. Total period spent for the heating process was 30 min. At the end of heating period, the crucible was removed, cooled in a desiccator and weighed.

Conradson carbon residue was calculated as

\[
\text{Conradson carbon residue (\%) } = \frac{A \times 100}{W}
\]

Where,

\[A = \text{Mass of carbon residue (g)}\]
\[W = \text{Mass of sample (g)}\]

**Sulphated ash**

Test for sulphated ash was made as per the procedure outlined by Gerpen et al. (2004b). A crucible was preheated to 775 °C in a furnace for a minimum period of 10 minutes. It was then removed, cooled in a desiccator and weighed \((W_1)\). Five gram of methyl ester was taken into it and was moistened with few drops of conc. sulphuric acid. The crucible was heated gently until fumes were evolved. When fuming ceases, the sample was heated more strongly and continuously until all the carbonaceous matter burned off. The crucible was allowed to cool. Again it was moistened with a few drops of conc. sulphuric acid. It was heated again more strongly until white fumes cease to be evolved. It was finally ignited in a muffle
furnace at 775 °C for 30 min. Crucible was then removed, cooled in a desiccator and weighed ($W_2$).

Sulphated ash was calculated as a percentage of the original sample,

$$\text{Sulphated ash (\%)} = \frac{W_2}{W_1} \times 100$$

*Where,*

$W_1$ = weight of the sample (g),

$W_2$ = weight of sulphated ash (g).

**Water and sediments**

This test was carried out as per the procedure given in ASTM D 1796 - 04. 10 ml of biodiesel was taken in each of two centrifuge tubes and centrifuged at 800 rpm for 10 minutes at 60 °C. After the centrifugation, the volume of water and sediments settled at the bottom of the centrifuge tube was assessed and expressed in percent.

$$\text{Water and sediments (\%)} = V_1 + V_2$$

*Where,*

$V_1$ and $V_2$ are the volume of water and sediment present in 10 ml of the sample contained in the respective tubes.

**Volutility**

Volatility of diesel and biodiesel was assessed as per the procedure provided in ASTM D 1160 - 06.
Flash point

The flash point of the methyl ester was tested as per the procedure contained in ASTM D 93 - 07.

Pour point

Pour point test was carried out as per the procedure narrated in ASTM D 97 – 07. Test jar with the cork carrying the high pour thermometer was taken. The thermometer bulb was immersed to 3 mm below the surface of the sample. The sample was heated without stirring to 45 °C in a bath maintained at 48 ± 1.5 °C. The test jar was then transferred to a bath maintained at 24 ± 1.5 °C. When the sample reached 27 °C, the jar and the high pour point thermometer was held in a horizontal position for 5 seconds and the movement of the sample in the jar was noticed. The experiment was repeated until the sample shows any movement. Test jar was then transferred to a cooling bath to allow the formation of paraffin wax. If the movement of sample in the test jar was noted, then the test jar was replaced immediately. The test was repeated at a next temperature, 3 °C lower.

Readings of the thermometer were taken and 3 °C was added to the temperature and recorded as a result. Three trials were conducted for each sample to check the consistency of the results.

Cloud point

The cloud point of the methyl ester was investigated as per the procedure described in ASTM D – 2500- 05 standard method.

Total glycerol content

AOCS ca 14 - 56 method was used to determine the total glycerol present in the finished product. In a 250 ml conical flask, 2 ml of the sample and 50 ml of
0.5 M alcoholic potassium hydroxide solution were taken. An air condenser was fitted to the same conical flask and the sample was gently boiled for 30 min. Parallely in a standard volumetric flask of 1 litre capacity 99 ml of chloroform was taken. 25 ml of glacial acetic acid was added to it. The conical flask was removed from the water bath and the content was transferred to the volumetric flask. The conical flask was rinsed with 500 ml distilled water and it was poured in the standard volumetric flask. The flask was stoppered and shaken vigorously for 30 to 60 seconds. Distilled water was added up to the mark and mixed thoroughly by inverting it. The flask along with the content was kept undisturbed until aqueous and chloroform layers were separated. Once the separation was completed, the aqueous layer was removed and the same was poured in the standard volumetric flask in which 50 ml was taken in a beaker. To this 50 ml of periodic acid reagent was added to it. Two blanks were prepared by adding 50 ml of distilled water to each beaker containing 50 ml of periodic acid reagent. Beakers were covered with watch glass and allowed to stand for 30 min. Before titrating the samples, 20 ml of 15 percent potassium iodide solution was added, mixed and allowed to stand for one minute. The sample was diluted with distilled water and the resultant content was titrated against 0.1 N sodium thiosulphate solutions. The titration was continued until brown iodine colour almost disappeared. Two ml starch indicator was then added to this and the titration was continued until blue iodine starch complex. The total glycerol content was calculated as follows:

\[
\text{Total glycerol content (\%) = } \frac{(B - S) \times N \times 2.302}{W}
\]

Where,

- \(S\) = Volume of sodium thio sulphate titrated to sample (ml).
- \(B\) = Volume of sodium thio sulphate titrated to blank (ml).
- \(N\) = Normality of sodium thio sulphate solution used.
- \(W\) = Weight of sample (g).
- 2.302 = molecular weight of glycerol / 40.
Free glycerol content

Free glycerol content of methyl esters was determined according to AOCS ca 15 - 46 method. 10 gram of the sample was taken in a conical flask. Using 90 ml of chloroform the whole content was washed and transferred to a one litre capacity volumetric flask. To this, 500 ml distilled water was added to it and the flask was vigorously shaken for one minute. The distilled water was then added up to the mark. It was stoppered and mixed thoroughly by inverting and setting aside until the aqueous and chloroform layers separated. The aqueous layer was removed and 100 ml of it was transferred to a beaker containing 50 ml of periodic acid reagent. Two blanks were prepared. Beakers were covered with watch glass and allowed for 30 min. Before titrating the sample, 20 ml of 15 % potassium iodide solution was added, mixed and allowed to stand for one minute. The sample was diluted with distilled water and the resultant compound was titrated against 0.1 N sodium thiosulphate solution, the titration was continued until the brown iodide colour almost disappeared. Two ml of starch indicator was added to this and the titration was continued until blue iodine starch complex colour just disappeared.

The free glycerol content was calculated using the equation

\[
\text{Free glycerol content (\%) = \frac{(B - S) \times N \times 2.302}{W}}
\]

Where,
- \(S\) = Volume of sodium thio sulphate titrated to sample (ml).
- \(B\) = Volume of sodium thio sulphate titrated to blank (ml).
- \(N\) = Normality of sodium thio sulphate solution used.
- \(W\) = Weight of sample (g).
- 2.302 = molecular weight of glycerol / 40.

Combined glycerol content

The combined glycerol content of the sample was calculated as the difference between the total glycerol content and free glycerol content in the test sample.
## Standards of biodiesel

The American Society of Testing and Materials (ASTM) have approved ASTM D 6751 a standard specification for biodiesel and are represented in Table 2. Biodiesel produced should meet the above requirement level.

<table>
<thead>
<tr>
<th>Property</th>
<th>ASTM Method (D6751)</th>
<th>Limits</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific gravity</td>
<td>D 1250 - 08</td>
<td>0.87 - 0.90</td>
<td>g/cm³</td>
</tr>
<tr>
<td>Kinematic viscosity at 40º C</td>
<td>D 445</td>
<td>1.9 - 6</td>
<td>cSt</td>
</tr>
<tr>
<td>Calorific value</td>
<td>D 240 - 02</td>
<td>-</td>
<td>MJ/Kg</td>
</tr>
<tr>
<td>Acid number</td>
<td>D 664</td>
<td>0.50 max</td>
<td>mg KOH/g</td>
</tr>
<tr>
<td>Iodine value</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Saponification value</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Peroxide value</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Sulphated ash</td>
<td>D 874</td>
<td>0.02</td>
<td>%</td>
</tr>
<tr>
<td>Sulphur</td>
<td>D 5453</td>
<td>0.05</td>
<td>%</td>
</tr>
<tr>
<td>Water &amp; Sediments</td>
<td>D 2709</td>
<td>0.05</td>
<td>%</td>
</tr>
<tr>
<td>Copper corrosion</td>
<td>D 130</td>
<td>No. 3 max</td>
<td></td>
</tr>
<tr>
<td>Vacuum distillation @ 90%</td>
<td>D 1160</td>
<td>360 max</td>
<td>%</td>
</tr>
<tr>
<td>Carbon residue</td>
<td>D 4530</td>
<td>0.05</td>
<td>%</td>
</tr>
<tr>
<td>Flash point</td>
<td>D 93</td>
<td>130 min</td>
<td>ºC</td>
</tr>
<tr>
<td>Cloud point</td>
<td>D 2500</td>
<td>Report</td>
<td>ºC</td>
</tr>
<tr>
<td>Pour point</td>
<td>D 97</td>
<td>Report</td>
<td>ºC</td>
</tr>
<tr>
<td>Cetane number</td>
<td>D 613</td>
<td>47 min</td>
<td>-</td>
</tr>
<tr>
<td>Total glycerol</td>
<td>D 6584</td>
<td>0.240 max</td>
<td>-</td>
</tr>
<tr>
<td>Free glycerol</td>
<td>D 6584</td>
<td>0.020 max</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 2: ASTM D 6751 specification for biodiesel
**Stability of biodiesel**

Long term storage tests on biodiesel were conducted for a period of 12 months. Samples were stored in glass containers at room temperature. At regular intervals (every month) the characteristics such as acid value, free fatty acids, peroxide value, kinematic viscosity, iodine value, specific gravity and water and accumulation of sediments of the stored biodiesel were analysed.