# BIODIESEL PRODUCTION FROM THE SEED KERNEL OF DESERT DATE FRUIT

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**ABSTRACT**

The *Balanites aegyptiaca* belongs to the family Balanitaceae, also called as desert date. It is a deep-rooted arid zone tree has a very wide natural range. The tree is valued for its fruits and seeds. It has a diverse use in treatment of diarrhoea, haemorrhoid, stomach aches, jaundice, yellow fever, syphilis and epilepsy. The fruit is used to treat liver disease and as a purgative. The seed kernel is rich in oil, protein and minerals. The seed kernel consists of 45-53 % of oil content in them. The fruits were shade dried and the fruit was broken to separate the seed kernel. A soxtherm extraction of seed kernel is done using the solvent petroleum ether and oil is extracted. The production of biodiesel was carried out using the extracted oil through transestrification method. The biodiesel parameters showed that its cloud point, pour point, flash point, viscosity, density, fire point, Saponification value, cetane index and iodine value were 7 cSt, 3 cSt, 165˚C, 4.796 cSt, 876.950kg/m3, 190 ˚C, 525.93, 54.773 and 8.466 respectively. The results of the biodiesel analyses were found to compare very well with the standard values, indicating that the extracted desert date seed oil was a good biodiesel with a yield of 88%.

**Key words:** Oil extraction, seed, Desert date fruit, Biodiesel**,** transesterification

## INTRODUCTION

The *Balanites aegyptiaca* (L.) Del (Desert Date) is a species of tree, classified as a member of the Balanitaceae family. This tree is native to Africa and parts of Middle East. In Kannada it is called as ingala kai, Ingudi. In India, it is particularly found in Rajasthan, Gujarat, Madhya Pradesh and Deccan. It can be found in many kinds of habitat, tolerating a wide variety of soil types, from sand to heavy clay and climatic moisture levels. The leaves are sub sessile, grey green in colour, obviate in shape and apex is acute. The fruit has thin brittle epicarp, a fleshy mesocarp and a woody endocarp containing the oily seed or kernel.

It has many traditional uses in the treatment of jaundice, intestinal worms, infection, malaria, syphilis, epilepsy, dysentery, constipation, diarrhoea, stomach aches, asthma and fever. The oil and fruit extracts of banalities were reported to have vast biological activities as anticancer, antihelmenthic, useful botanical insecticides, antifungal and molluscicidal activities. It has hypertensive, antibacterial, antifungal, antiviral, depressant, hepatoprotective, anti-inflammatory, anticancer, antibiotic, stimulant, antitubercular, antifertility action and anticancer activity. The leaves are anti-inflammatory, antihelmenthic, ophthalmic, rich in vitamin A & C. They are useful in scurvy, wounds, tumours, inflammations, and helminthiasis. The seed oil of kernel of *Ingudi* is used in treatment of skin diseases, burn, excoriation, chronic vitiated ulcers & freckles & is indicated from *Nighantu* period. But significant work has not been done so far on *Ingudi* leaves. Leaf is also a potent antimicrobial agent action known from ancient times. They have wound healing & antimicrobial activity with specific bacterial strains which are causative organism for surgical wound infections (Manji, 2013).

The biodiesel, is an alternative fuel, has been currently receiving much attention owing to the limited availability of conventional petroleum diesel and environmental concerns. Bio diesel is a renewable energy source that has superior properties than that of petro-diesel fuel such as nontoxicity. The research involving the production of fatty acid methyl esters are being embarked on nowadays because it is very important for today's world to identify an alternative to fossil fuel to meet the future demands for energy based on the fact that diesel fossil fuel reserves dwindling and at a time will run out, especially for use in internal combustion engines, which reduce the peak flame temperature and thereby reduction in various emissions. The bio diesel is an alternative fuel obtained from renewable biological sources such as vegetable oils and animal fats. It can be directly used to replace petroleum diesel without modifying diesel engines since their properties, e.g., specific gravity, cetane number, viscosity, cloud point, and flash point, are similar. It is a promising alternative to conventional petroleum-based diesel fuel. Furthermore, it has a number of advantages such as reducing carbon dioxide emission by about 78%, nontoxicity and biodegradability. These benefits have made the fuel a very good environmentally benign one (Akaagerger *et al.*, 2016).

**Methodology**

### Sample collection and preparation

The desert date fruit used in this study were collected from Bagalkot district of Karnataka, India. After the fruits were obtained, they were washed and shade dried and their shells were cracked using hammer to separate its seed kernel (Figure 1). The kernels of the dried seeds were ground using mortar and pestle and then separated with the aid of a sieve shaker.


**Figure 1. Raw desert date fruits and seed kernels of desert date fruits**

**Oil Extraction**

The oil content in seeds was determined using soxtherm apparatus which works on the principle of solvent extraction. The sun-dried seeds were powdered in mixture grinder. Powdered seed samples were taken in a cotton thimble and plugged with cotton and placed in pre-weighed soxtherm jars containing boiling stones. About 100 ml petroleum ether was added to these jars and placed in soxtherm apparatus. The oil was extracted by running the pre-programmed soxtherm apparatus for 4 h 35 min. After completion of extraction, the remaining petroleum ether and moisture was removed by keeping it in hot air oven at 110 ˚ for 1hr. The jars were removed from the oven and placed in desiccators containing calcium carbonate for one hour to remove moisture. The oil content in the seeds was determined using following formula,

Oil content (%) = $W_1$- $W_2$/W×100

Where W, weight of powdered seed sample; $W_1$, weight of soxtherm jar along with boiling stones; $W_2$, final weight of soxtherm jar along with boiling stones and extracted oil (Rajesh *et al.*, 2012).

**BIODIESEL PRODUCTION FROM OIL**

The extracted oil was then filtered and used for Biodiesel production. The biodiesel is produced from vegetable oil or animal fat. The primary constituent of vegetable oil is a mixture of triglycerides. Transesterification of triglycerides using anhydrous alcohol in the presence of a strong base like sodium hydroxide as catalyst yields fatty acid mono -alkyl ester (FAME) which is known as biodiesel and glycerine as a by-product.

**Pre- treatment for high FFA oil**

Oils are glycerol esters of fatty acids, molecules made of glycerine and fatty acids. These triglycerides are oxidized into free fatty acid (FAA) during storage. These free fatty acids form soap during Transesterification process. If the FAA is more than 3% the recovery of biodiesel is reduced drastically. Hence, an acid pre-treatment is given during which FFAs are converted into biodiesel by acid- esterification. Vegetable oils having low free fatty acid content (below 3%) can be converted into biodiesel by direct Transesterification. The acid esterification was carried out using concentrated sulphuric acid as catalyst. Conc. $H_2SO_4$ at a rate of 5% of FFA (0.05 g $H_2SO_4$ for every 1.0g of FFA) was added to methanol in 40:1methanol to FFA molar ratio.

**Procedure**

The weight of FFA in one liter was determined based on the acid value. The volume $H_2SO_4$ (mL) required was determined by (Weight FFA× 0.05)/ Density of $H_2SO_4$.

The number of moles of FFA in the oil sample was determined based on the average molecular weight of fatty acids. The volume of methanol required was calculated using 40:1 mole ratio of methanol: FFA. The methanol-$H_2SO_4$ mixture was prepared by adding conc. $H_2SO_4$ drop wise into methanol. The oil was preheated to 60 ˚C in a three-neck flask fitted with condenser, temperature sensor and catalyst dozer. The methanol- $H_2SO_4$ mixture was added slowly and allowed to react for the 1 h. After the completion of the reaction the mixture was kept settling for an hour which formed a thin upper acid rich layer. The upper layer was discarded and the lower layer was tested for FFA content. The lower layer was then subjected to Transesterification process. The amount of catalyst required was determined by titration (Rajesh *et al.*, 2012).

**Titration testing**

The amount of NaOH required to neutralize the FFA present in the oil was determined by titrating testing using known concentration NaOH solution. Generally, the oil without FFA requires 3.5g NaOH for transestrification. Extra amount of NaOH need to be added to neutralize FFA which consequently formed into soap.

**Procedure**

One gram of oil was dissolved in 10mL of anhydrous isopropyl alcohol in a 100 mL conical flask. The sample was titrated against sodium hydroxide solution using phenolphthalein indicator. Amount of NaOH in mL required to neutralize the sample gives, grams of NaOH required to neutralize FFA present in one litre of oil.

| | | |
|---|---|---|
| Amount of NaOH required for Transesterification of Triglycerides (3.5g) | + | Amount of NaOH required for neutralization of FFA (titration) |

**Transesterification process**

**Procedure**

500ml of oil was taken in a one liter three necked flask fitted with a reflux condenser, catalyst dozer and temperature sensor. The oil was heated to required temperature on a magnetic stirrer with heating controller. Sodium meth oxide was added slowly to the pre- heated oil with stirring. The Transesterification reaction was carried out for 2hrs. Then it was subjected for settling in a separating funnel to form upper biodiesel layer and lower glycerine layer. Glycerine was drained out. The bio diesel thus produced was washed two times with equal quantity of water acidified with acetic acid (0.1%) to avoid emulsification. Then again washed two times with water to remove the soluble contaminants. Bio diesel was then dried by heating at 110˚C till the moisture content was removed completely. It was cooled and filtered, then subjected for future analysis (Rajesh *et al.*, 2012).

## QUALITY ASSESSMENT OF BIO DIESEL

**Determination of kinematic viscosity of biodiesel**

The resistance to flow of a fluid under gravity is known as kinematic viscosity.

Kinematic viscosity of bio diesel was determined using calibrated Cannon -Fenske viscometer as per ASTM D44 **(**Kumar *et al.*, 2013).

**Procedure**

The viscosity of bio diesel sample was carried out using Cannon- Fenske viscometer No.100 with nominal constant 0.015.

**Determination of Density of Oil**

The density of the oil and biodiesel was determined by the pycnometry.

**Procedure**

The empty weight of pycnometer ($W_1$g) was noted. The pycnometer was filled with the distilled water and the weight ($W_2$ g) was noted. The pycnometer was emptied and dried, and filled with the sample and the weight ($W_3$ g) was determined **(**Kumar *et al.*, 2013).

Calculation:

$$\text{Density} = \frac{(W_3-W_1) \times 1000 \text{ Kg/m}^3}{(W_2-W_1)}$$

**Determination of Iodine Value of Biodiesel**

Iodine number of the oil is a measure of the degree of un-saturation present in oil. It is expressed as a number of grams of iodine absorbed by 100 grams of oil. The excess of iodine remaining is estimated by titrating against sodium thiosulphate (Ameen *et al.*, 2011).

**Procedure:**

Transfer exactly 0.3g of oil to a clean dry iodine glass containing 10mL of chloroform /carbon tetrachloride and mixed well. Twenty-five millilitre of IBr was added to it. One conical flask was kept as a blank without oil. All the flasks were shaken well and kept in dark. Stirring was done for each flask for every 5 min for about 30 minutes. 50 millilitres of water was added to each flask.10 millilitre of 10% KI to reach flask was added. The solutions were titrated against standard $Na_2S_2O_3$ solution using 2mL of starch as indicator with end point blue to colourless. Iodine number was calculated by using the following formula,

$$\text{Iodine value} = \frac{[V_1-V_2] \times N \times 126.9 \times 100}{W \times 1000}$$

Where, $V_1$, volume of $Na_2S_2O_3$ used for blank; $V_2$, volume of $Na_2S_2O_3$ used for oil; N, normality of $Na_2S_2O_3$; W, weight of oil

**Determination of saponification value of Biodiesel**

When the oil is heated with KOH, it is saponified and releases fatty acids and glycerol. The fatty acids neutralize the sodium hydroxide and the above titration detects the amount of alkali that has been used for saponification. Each mole of triglycerides uses 3 moles of KOH for saponification (Singh *et al.*, 2009).

**Procedure**

One gram of oil was added into a 250Ml clean round bottomed flask.10 mL of ethanol/ether mixture (2:1 v/v) and exactly 25mL of 0.5 N alcoholic KOH were added. The mixture was refluxed for one hour. Then the contents were cooled down. Simultaneously, another flask without adding the oil was processed as blank. The solutions were titrated against 0.5N HCI using phenolphthalein indicator.

$$\text{Saponification value} = \frac{56.1 (V_1 - V_2) \times N}{\text{Weight of oil}}$$

Where, $V_1$, volume of $Na_2S_2O_3$ used for blank; $V_2$, volume of $Na_2S_2O_3$ used for oil; N, normality of $Na_2S_2O_3$; W, weight of oil.

**Determination of cloud point and pour point of biodiesel (**Ferdous *et al.*, 2013)

**Cloud point**

Cloud point is a test used to characterize the low temperature performed of bio diesel fuel. It defines the temperature at which a cloud or haze appears in the fuel under prescribed test condition. Cloud point of biodiesel was determined as per ASTM D2500.

**Procedure**

The filtered bio diesel sample was poured into the test jar to the level mark. The test jar was closed tightly by the cork carrying the test thermometer with the thermometer bulb resting on the bottom of the jar. A disk was placed at the bottom of the jacket 10 min before the test jar was inserted. A gasket was placed around the test jar, 25 mm from the bottom. The test jar was placed inside the jacket. The temperature of the cooling bath was maintained at 0±1.5˚C by stuffing freezing mixture containing crushed ice and sodium chloride in the cooling bath around the jacket. The specimen was inspected for cloud at intervals of 1˚C and replaced in the jacket within 3sec. The cloud point was noted, at which any cloud is observed at the bottom of the test jar.

**Pour point**

The temperature at which the biodiesel ceases to flow is known as pour point. The pour point of biodiesel was determined as per ASTM D97.

**Procedure**

The filtered bio diesel sample was poured into the test jar to the level mark. The test jar was closed tightly by the cork carrying the test thermometer with the thermometer bulb resting on the bottom of the jar. A disk was placed at the bottom of the jacket 10 min before the test jar was inserted. A gasket was placed around the test jar, 25 mm from the bottom. The test jar was placed inside the jacket. The temperature of the cooling bath was maintained at 0±1.5˚C by stuffing freezing mixture containing crushed ice and sodium chloride in the cooling bath around the jacket. The specimen was inspected for cloud at intervals of 1˚C and replaced in the jacket within 3sec. The pour point was noted, at which the sample ceased to flow is observed at the bottom of the test jar.

**Determination of calculated cetane index**

The physical and chemical properties of fuel play very important role in delay period. The cetane number of the fuel is one such important parameter which is responsible for the delay period. Cetane index was calculated based on the iodine value and saponification value according to Krisnangkura (1986) using the following relationship (Rajesh *et al.*, 2012)

Calculated cetane index = 46.3 + (5458/SV) – (0.225 × IV)

Where; SV, Saponification value; IV, Iodine value

**Determination of Moisture content**

The moisture content was calculated by the procedure given by AOAC (2012), taking glass beaker of 50ml. The beaker was weighed in weighing machine. About 5ml of biodiesel was poured in the beaker. The beaker was weighed again and kept in hot air oven at 60 for 24hour. The beaker was allowed to cool and then weight is taken (Babagana, 2011).

Moisture content = $(W_2\text{-}W_3)/W_1 \times 100$

W1 is the initial weight of empty beaker,
W2 is the initial weight with biodiesel sample, W3 is the final weight

**Determination of Flash point and Pour point**

Flash point is the lowest temperature corrected to a barometric pressure of 101.3kPa, at which application of an ignition source causes the vapours of a specimen of the sample to ignite under specified conditions of test. The flash of biodiesel was determined using Pensky – Martens closed cup apparatus as per ASTM D93.

**Procedure**

The test cup was filled with the biodiesel test sample to the filling mark inside the test cup. The test cover was placed on the test cup and the assembly was placed into the apparatus. The thermometer (IP 15C) was inserted into the holder. The test flame was ignited. The electric heater was switched on and the temperature raise was adjusted to 5- 6˚C/min. The sample was stirred at a speed of 100-120 rpm. The test flame was applied at an interval of 2˚C raise in temperature after reaching the temperature of the sample to 90˚C. The test flame was applied by lowering the test flame using the shutter operator on the test cover. The temperature at which the test flame, when lowered into the vapor space, caused a blue halo and then a distinct flash in the interior of the test cup was noted as the flash point. The temperature at which biodiesel ignites and continues to burn at least 5minutes, is noted as fire point (Ravikumar *et al.*, 2011).

**RESULTS AND DISCUSSION**

Table.1 Showing the results and compared values of biodiesel

| Properties | Biodiesel values | Standard Biodiesel values | Diesel standard values |
|---|---|---|---|
| Density (Kg/m$^{3)}$ | 876.950 | 860-900 | 820-950 |
| Cloud point (cSt) | 7 | -3to12 | -15 to 5 |
| Pour point (cSt) | 3 | -15 to 10 | -35 to -15 |
| Viscosity (cSt) | 4.796 | 1.9-6.0 | 1.3 - 4.1 |
| Saponification value | 525.93 | - | - |
| Iodine value | 8.466 | 126 max | - |
| Cetane index | 54.773 | 47-67 | 50 |
| Moisture content (%) | 0.03 | 0.05 | 0.161 |
| Flash point (˚C) | 165 | 190 | 52-96 |
| Fire point (˚C) | 190 | - | - |

The desert date seed kernel constitutes 53 % of oil content and biodiesel yield is 88 %. The seed had low moisture content, which was an indication of good shelf life because there is little or no water for hydrolysis to takes place. The values are close to those reported in Bauchi, Nigeria where (Akaagerger *et al.*, 2016), these oil yield from the kernels was found to be 45.2 % and biodiesel yield was found to be 82%. The oil yield was found to be 49.9 % (Manji *et al.*, 2013) indicating that the oil content is high meaning that oil percentage differs due to the place of collection and time of collection. Since high percentage of the oil is considered to be a good source for various purposes. This would ultimately help to domesticate this highly potential but most neglected plant in arid regions of Africa and Asia, which could certainly help to improve the ecological and financial situation of the world's most difficult area. The edible fruits are rich in saturated fatty acids which are used as cooking oil. It has virtually no Sulphur content, better lubricating properties and higher cetane number than diesel. It can be used in blended form with diesel at any proportion for use in an internal combustion engine or burners.

The cetane number is the period between the start of injection and the first identifiable pressure increases during combustion of the fuel. Fuels with higher cetane number will combust more easily in diesel engine. Biodiesel has a higher cetane number compared to diesel as it contains oxygen. The chemical and physical properties that were studied are following: kinematic viscosity, flash point, pour point density, specific gravity, saponification value, iodine value and acid value. Fatty acid composition and physical and chemical properties of the desert date biodiesel were also determined and compared with standard values of diesel and biodiesel is showed in (Table.1) Ferdous *et al.*, (2013).

The viscosity is a measure of the internal friction or resistance of oil to flow. As the temperature of oil is increased, its viscosity decreases and it is therefore able to flow more readily. Viscosity is the most important of biodiesel since it affects the operation of fuel injection equipment, particularly at low temperatures when the increase in viscosity affects the fluidity of the fuel. High viscosity leads to poorer atomization of the fuel spray and less accurate operation of the fuel injectors. The viscosity values of vegetable oils are between 27.84 and 52.76 mm$^2$/ sec at 40°C whereas those of vegetable oils methyl esters are between 3.6 and 5.7 mm$^2$/sec. Non-edible oils were observed to have high viscosity values about six times more than ASTM limits. The Jatropa was the most viscous with a viscosity of 52.76 mm$^2$/sec at 40°C. The methyl esters and the blends were observed to have viscosity within the ASTM limits. The value of desert date is showed in (Table.1) (Ravikumar *et al.*, 2011).

The density is another important property of biodiesel. The biodiesel of sun flower oil has a minimum density value of 0.86 kg/l. The diesel fuel sample was observed to have a density value of 0.836 kg/l which is lower than edible, non-edible oils, methyl esters and its blends. The flash point of vegetable oil methyl esters is much lower than those of vegetable oils. The flash point of sun flower oil was observed to be highest 274°C among methyl esters rape seed was observed to be lowest with 80°C, the result of desert date biodiesel is showed in (Table.1) (Ravikumar *et al.*, 2011).

**CONCLUSION**

The results obtained from desert date seed oil was that it can be used economically as a viable oil source because its oil content was found to be high. Also, the oil parameters showed that the oil was composed of moderately long chain fatty acids. The free fatty acid profile gave the different composition of the oil. Oleic acid was found to be high it reduces blood pressure, prevents ulcerative colitis, increases fat burning to help with weight loss, protects cells from free radical damage and generates brain myelin.

Stearic acid is mainly used in the production of detergents, soaps, and cosmetics and personal care products it acts as a skin cleanser. Palmitic acid is used in cosmetics, personal care products. Linoleic acid is necessary for skin and hair growth, energy production, healthy brain function, bone density and reproductive health. The parameters also helped to conclude that oil sample had less amount of fatty acid like linoleic acid. The free fatty acid profile helps to conclude on the quality of the oil from nutritional stand point. Further investigation, like the nutrition content, will help in better conclusion regarding the nature of the oil.

Due to these properties the oil, it is concluded that it can be used as a cleansing agent and used in cosmetics, as well as cooking oil and pharmaceutical companies. More than 80%of people living in urban areas that monitor air pollution are exposed to air quality levels that exceed the world health organization limits. The vegetable oils are extremely viscous with viscosities ranging from 9 to 16 times greater than petroleum diesel. Biodiesel an alternate diesel fuel is made from renewable biological sources such as vegetable oils and animal fats by transestrification reaction using methanol. The purpose of the transestrification process is to lower the viscosity of the oil.

As urban air quality declines, the risk of stroke, heart disease, lung cancer and chronic and acute respiratory diseases, including asthma. It reduces acid rain. Acid rain results when sulphur dioxide and nitrogen oxide are emitted into the atmosphere. Bio Diesel produces 80% less carbon dioxide and 100% less sulphur dioxide emissions. It provides a 90% reduction in cancer risk. Biodiesel is a sulphur free fuel. Acid rain may cause harmful effects on soils, forests, streams and lakes. The biodiesel also reduces global warming.

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