

# Research Article STUDIES ON PROCESS OPTIMIZATION FOR BIODIESEL PRODUCTION FROM NON-EDIBLE OILS

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Abstract: Biodiesel can be produced from vegetable oils by transesterification process. The fatty acid composition of oils can be varied from oil crops to tree borne oilseeds. It is essential to find the optimal conditions for maximum methyl ester conversion for each biodiesel feedstock. This paper describes the biodiesel production and optimizes the process conditions for three non-edible oils viz, pongamia, jatropha, and neem oils. The optimal conditions for best yield of methyl ester from jatropha and neem oil were observed with a molar ratio of 1:6, catalyst amount of 0.3 M and reaction time of 2 h. The molar ratio of 1:7.5, catalyst amount of 0.3 M and a reaction time of 2 h were found to be optimal conditions for methyl ester production from pongamia oil. Under optimized conditions, the maximum methyl ester conversion was found as 99.70, 98.88 and 97.65 % for biodiesel production from jatropha, pongamia and neem oils respectively. The calorific value for this methyl ester was ranged from 38.11 to 39.81 MJ/kg. The kinematic viscosity of methyl ester from pongamia, jatropha and neem oils at 40°C was found to be 1/7.5, 1/6.6 and 1/7.6<sup>th</sup> of the viscosity of raw oil respectively. The flash point, cloud point and pour point of these methyl esters were found to be within the permissible range of ASTM standards.

Keywords: Transesterification, Pongamia Methyl Ester, Jatropha Methyl Ester, Neem Methyl Ester

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## Introduction

Petroleum fuels is playing an essential role in the development of industrial growth, transportation; agriculture and also it fulfils the energy requirement for other basic human needs. Moreover, their alarming usage in different applications would create many environmental problems. Furthermore, the increase in crude oil import affects the country's economy, and ever-increasing fuel's cost has led to search the alternative biofuels for ensuring energy sustainability, security and also mitigating climate change. Since the petroleum reserves limited, biofuels can provide a viable solution to reduce dependence on oil imports for the developing countries. Biodiesel is recommended alternate biofuel to replace the diesel fuel, which can be used in diesel engines without any modifications. Generally, edible oils such as cottonseed, palm, sunflower, rapeseed, and safflower can be used for biodiesel production and found satisfactory results for usage of blended or 100 % biodiesel as fuel in the diesel fuelled engines. There is huge consumer demand for these edible oils for food consumption in India. So, biodiesel research is focused on non-edible oils to overcome this food vs. fuel conflict. The non-edible oils such as castor, rice bran, linseed, Pongamia (Pongamia glabra), Jatropha (Jatropha curcas L) and Neem (Azadirachta indica), madhuca (Madhuca indica), rubber (Hevea brasiliensis), etc. are available in larger quantities and unexploited feedstocks for biodiesel production. As similar to edible vegetable oils, these oils cannot be used as fuel in diesel engines due to their poor combustion characteristics, higher kinematic viscosity, and density. Generally, vegetable oils or animal fats are used to produce biodiesel through transesterification process [1, 2]. Biodiesel quality depends on the fatty acid compositions of the oils. The methyl ester conversion and process conditions for biodiesel production would be varied for different biodiesel feedstocks. This paper is examined how process conditions

affect the methyl ester conversion for biodiesel production from three non-edible oils *viz.*, pongamia, jatropha and neem oils, and experimental results are briefly discussed in this paper.

## Materials and methods

The essential fuel properties of raw non edible oils and their biodiesels were determined by using the ASTM and AOCS methods *viz.*, calorific value (IS: 1448-1960), kinematic viscosity (ASTM 445-72), specific gravity (IS: 1448-1972), flashpoint (IS: 1448-1992), cloud and pour point (ASTM D-97/57), carbon residue (ASTM D524-IP14/65), ash content (IS: 1448-1992), free fatty acid and acid value (AOCS Ca 5a-40), iodine value (AOCS Cd 1c-85) and saponification value (AOCS: Cd 3-25).

## **Experimental details**

Generally, the molar ratio and catalyst amount are more influential parameters on methyl ester conversion. In order to determine best optimal conditions for maximum methyl ester conversion, three levels of molar ratio (1:4.5, 1:6 and 1:7.5), three levels of catalyst amount (0.1, 0.2 and 0.3 M) and 2 h reaction time were used in the biodiesel experiments. For this study, methanol and sodium hydroxide were used for biodiesel production. The sodium methoxide solution was prepared by mixing the appropriate amount of methanol and sodium hydroxide. This chemical solution was added to oil and stirred in a three neck laboratory scale reactor for 2 h at 60°C. The reactants were poured into separating funnel after completion of reaction time and allowed for gravity settling for 12 h to separate the glycerol from biodiesel.

International Journal of Agriculture Sciences ISSN: 0975-3710&E-ISSN: 0975-9107, Volume 11, Issue 6, 2019 Glycerol settled at the bottom, and the crude biodiesel above the glycerol layer was collected. The collected biodiesel was washed three times with water, and the glycerol content in biodiesel was determined by A.O.C.S Ca14-56 method. The methyl ester conversion was calculated by following formula based on

glycerol content in the raw oil and biodiesel. Difference in glycerol content  $Methyl \ ester \ conversion \ \% \ = \frac{for \ raw \ oil \ and \ its \ biodiesel}{Glycerol \ content \ in \ the \ raw \ oil} \times 100$ 

## **Results and Discussions**

## Optimal conditions for biodiesel production

For this study, three levels of molar ratios and catalyst amount were used to determine the optimal conditions for higher methyl ester conversion in biodiesel production.

#### Pongamia biodiesel production

At 1:4.5 molar ratio of oil to methanol, the methyl ester conversion increased from 82.63 to 89.50% [Fig-1] as the catalyst amount increased from 0.1 to 0.3 M. At 1:6 molar ratios, the methyl ester conversion raised from 91.20 to 94.73% as catalyst amount increased from 0.1 to 0.3 M. For 1:7.5 molar ratio, the methyl ester conversion increased with molar ratio as well as with catalyst amount. The best conversion occurred at a molar ratio of 1:7.5 with 0.3 M catalyst amount.



Fig-1 Effect of molar ratio and catalyst amount on pongamia biodiesel production

#### Jatropha biodiesel production

The methyl ester conversion at a molar ratio of 1:4.5 increased from 95.30 to 97.43% due to an increase in catalyst amount from 0.1 to 0.2 M [Fig-2]. The methyl ester conversion for 1:6 molar ratios was recorded as 97.50, 98.37 and 99.53 for the catalyst amount 0.1, 0.2 and 0.3 M respectively. For 1:7.5 molar ratio, the methyl ester conversion increased with an excess amount of methanol and catalyst. The percent conversion for jatropha biodiesel was in closer range for molar ratio 1:7.5. In the present study, the higher methyl ester conversion was observed for the molar ratio of 1:7.5, but the molar ratio of 1:6 with catalyst 0.3 M and 1:7.5 molar ratio with 0.2 M were found to be same. The optimal conditions for jatropha biodiesel production were identified as a molar ratio of 1:6 with 0.3 M catalyst amount.



Fig-2 Effect of molar ratio and catalyst amount on jatropha biodiesel production

#### Neem biodiesel production

The higher catalyst combinations performed better than the other catalyst levels because the FFA content of neem oil was 5.60% [Fig-3]. The methyl ester conversion of the neem biodiesel at 0.1M catalyst amount was low due to an insufficient amount of the catalyst used to convert the FFA present in neem oil into biodiesel. The conversion at molar ratios of 1:6 and 1:7.5 with 0.3M was found to be 4.76 to 2.98% higher than that of 0.1M catalyst amount. The best conversion for neem oil into biodiesel was observed at a molar ratio of 1:6 with 0.3M catalyst amount.





#### Properties of raw oils and their biodiesel

The properties of produced pongamia jatropha and neem biodiesel are compared with ASTM, Indian Standards (BIS) and EN standards and presented in [Table-1].

## Calorific value

Calorific values of biodiesel were slightly lower than raw oils due to chemical changes after transesterification process in fatty acid compositions of oils. The heat contents of biodiesel were approximately 10 to 14% lower than that of diesel. The maximum value was observed for pongamia biodiesel (39.81 MJ/kg) and low value for neem biodiesel (38.11MJ/kg).

#### Kinematic viscosity

The kinematic viscosity of pongamia oil was reduced from 41.89 to 5.59 mm2/s after transesterification. The kinematic viscosity of pongamia biodiesel was found to be 1.15 times higher than that of diesel fuel (4.86 mm<sup>2</sup>/s). The kinematic viscosity of jatropha biodiesel was found to be 5.32 mm<sup>2</sup>/sec, whereas the viscosity of jatropha oil was 35.25 mm<sup>2</sup>/s. The viscosity of neem biodiesel found to be 13% reduction as compared with the viscosity of raw oil. Among the biodiesels produced, neem biodiesel recorded the highest viscosity (5.68 mm2/s). Jatropha biodiesel was found to be closer to that of diesel fuel (4.86 mm<sup>2</sup>/s); these biodiesels can be used in the diesel engine. The kinematic viscosity of these biodiesels was found within the requirements of DIN standards.

#### Specific gravity

The specific gravity of biodiesels varied from 0.8712 to 0.8739 at 40°C, which was comparable to DIN standards (0.875 to 0.890). After transesterification, the specific gravity of biodiesel was lower than that of oils due to the removal of glycerol from oils. Neem biodiesel recorded a higher specific gravity (0.8739) than other biodiesels, and the minimum specific gravity was recorded for jatropha biodiesel (0.8712) [3]. The recommended specific gravity for diesel fuel was 0.82 to 0.86 at 15°C (IS 1460: 2000).

#### Flashpoint

Flashpoint of biodiesels ranged from 2.6 to 3.5 times higher as compared with diesel fuel (51°C). The flash point ranged from 163 to 179°C and 218 to 251°C for biodiesels and their raw oil respectively. The lower flash point temperature of biodiesel indicates an improvement in the volatile property of the biodiesel. This might be due to the replacement of the glycerol by molecules of methanol in the

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## Sriramajayam, S., Ramesh D., Palaniselvam V., Kamaraj A. and Kamaraj S.

Table-1 Comparison of	properties of biodiesel with different standards

Property	Pongamia	Pongamia	Jatropha	Jatropha	Neem	Neem	Biodiesel standards		
	Ōil	Biodiesel	Oil	biodiesel	Oil	Biodiesel	ASTM	DIN 51606	BIS 15607: 2005
Calorific value, MJ/kg	40.00	39.81	39.73	39.17	39.78	38.11	-	-	-
Kinematic viscosity at 40°C, mm <sup>2</sup> /s	41.89	5.59	35.25	5.32	43.43	5.68	1.9-6.0	3.5-5.0	2.5-6.0
Specific gravity	0.9102	0.8714	0.9098	0.8712	0.917	0.8739	0.88	0.875-0.890	-
Flashpoint, °C	228	163	251	174	218	179	130 min.	>110	120 min.
Cloud point, °C	15	14	13	12	14	14	-3 to 2	-	-
Pour point, °C	2	1	4	3	1	-1	-15 to 10	-	-
Carbon residue, %	0.51	0.25	0.54	0.24	0.52	0.32	<0.50	<0.5	-
Ash content, %	0.017	0.012	0.013	0.011	0.019	0.014	-	-	-
Free Fatty Acids, %	4.40	1.1	3.01	0.90	5.60	1.2	-	-	-
Acid value	8.756	2.189	5.97	1.791	11.144	2.87	0.8	0.5	0.5 max
lodine value	90.0	92.5	101.2	104.1	75.6	77.2			
Saponification value	194.6	191.8	195.2	192.2	203.4	198.5			

fatty acid compositions of the biodiesel. The pongamia biodiesel recorded the lowest flash point temperature (163°C) and maximum value found for neem biodiesel (179°C). All the biodiesels recorded higher flash point than diesel and it meets the IS standards for biodiesels.

#### Cloud point

The cloud point of biodiesels was observed to be between 12 and 14°C, and it was slightly higher than that of raw oils, which may be due to changes in the composition of oils and biodiesels. The observed value of the diesel cloud point was 13°C. The cloud point of jatropha biodiesel was found 1°C lower than diesel. The pongamia and neem biodiesels recorded the same cloud point value (14°C), which indicated the poor cold flow properties due to crystals formation in biodiesel at low temperature. This biodiesel required additional heating during winter to improve the flow behaviour of the biodiesel.

#### Pour point

The lowest pour point was observed for neem biodiesel (-1°C), which was 2°C lower than diesel fuel. Jatropha biodiesel had a pour point of 3°C, which was 4°C for raw jatropha oil. The decrease in pour point of biodiesel may be due to variation in the fatty acid compositions.

#### Carbon residue

Carbon residue is an important property for indicating the coking characteristics of the fuel. The carbon residue for biodiesel must be lower than 0.50% (DIN Standards) for better engine performance. Carbon residue of biodiesels varied between 0.24 to 0.32%. This range was found to be within the acceptable range of biodiesel standards. The lowest carbon residue was recorded for jatropha biodiesel (0.24%), which was 0.51% for raw jatropha oil. Reduction in carbon residue indicates an improvement in coking characteristics of the fuel. The maximum carbon residue recorded for neem biodiesel was 0.32%. All the biodiesels produced in this study would meet the BIS standards.

#### Ash content

Ash content of biodiesels varied between 0.011 and 0.014% whereas it was 0.010% for diesel fuel. The ash content of jatropha oil was found as 0.013%, which reduced to 0.011% after transesterification. The reduction in ash content was due to change made in compositions after the reaction and glycerol removal. The lowest ash content was observed for jatropha biodiesel, and the highest value found for neem biodiesel.

#### **Chemical properties**

The chemical properties of biodiesels reveal impurity present in the biodiesels. The chemical properties studied for biodiesel are discussed below.

## Free fatty acids

The free fatty acids of biodiesel ranged from 0.9 to 1.20% for jatropha to neem biodiesel. The free fatty acid of their raw oils varied from 3.01 to 5.60%. The free fatty acid of biodiesel was lower than raw oils. This may be due to neutralization of free fatty acids by an excess amount of NaOH catalyst during the biodiesel

production process.

#### Acid value

The acid value of biodiesels was lower than DIN biodiesel standards. The maximum acid value was obtained for neem biodiesel (2.87) and minimum for jatropha biodiesel (1.791).

#### lodine value

The iodine value of biodiesel varied between 77.2 and 104.1. The jatropha biodiesel had a higher iodine value of 104.1. The lowest iodine value (77.2) was recorded for neem biodiesel.

#### Saponification value

Saponification value in case of diesel fuel was zero as it has no fatty acid. The saponification value of the biodiesels was varied between 191.8 and 198.5. The maximum saponification value was observed for neem biodiesel (198.5) and minimum value for pongamia biodiesel (191.8).

## Conclusion

A maximum methyl ester conversion for biodiesel production was 99.70, 98.88 and 97.65 % for jatropha, pongamia and neem oils respectively. The optimal conditions for catalyst amount and reaction time were found as 0.3 M, and 2 h for higher methyl ester conversion for all the oils tested for biodiesel production. The optimal for molar oil to methanol ratio was found at 1:6.75, 1:6 and 1:7.5 for biodiesel production from pongamia, jatropha and neem oils. It concluded that all the biodiesels produced from three non-edible oils were found be within the permissible range of different international and Indian biodiesel standards.

Application of research: Renewable fuel for replacing conventional fuel

Research Category: Bioenergy, Biofuel

Abbreviations: AOCS -The American Oil Chemists' Society, ASTM - American Society for Testing and Materials

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Conflict of Interest: None declared

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