Indian Journal of Science and Technology, *Vol 11(28), DOI: 10.17485/ijst/2018/v11i28/130780,* July 2018 *ISSN (Print) : 0974-6846*
ISSN (Online) : 0974-5645

Production of Methyl Ester of Mahua (Madhuca Biodiesel) for Improving its Cold Flow Characteristics

Amandeep Yadav* and Navdeep Sharma Dugala

Department of Automobile Engineering, Chandigarh University, Gharuan - 140413, Punjab, India;
amanyadav1311@gmail.com, navdeepdugala@gmail.com

Abstract

Background: In present day scenario, energy sector of developing world is depending upon fossil fuels. To reduce this dependency on fossil fuels, new alternative sources of energy must be developed. Opting for biodiesels can be an effective approach to replace these fossil fuels. **Methods**: Mahua oil is used to produce the methyl ester of Mahua (Mahua biodiesel) through transestrification process. These experiments were performed for the production of Mahua biodiesel from Mahua seed oil with methanol and catalyst of KOH. **Findings:** The Mahua biodiesel fuel properties are compared to the requirements of American (ASTM) standards for biodiesels. Also to improve the cold flow properties of Mahua biodiesel, it was mixed with the blends of Ethanol. **Improvements:** Blend of 20% Ethanol with Mahua biodiesel shows better pour point and cloud point than other samples.

Keywords: Cloud Point, Ethanol, Madhuca Biodiesel, Methanol, Pour Point,Transestrification

1. Introduction

Biodiesels are increasing their attention in the world as an aleternative fuel in place of fuel based on fossil fuel. Also it provides the low cost fuel, energy dependency to the countries and reduce the globle warming problems with the fossil fuels. The term biodiesel means the fuel which is produced by vegetable oils, animal fats etc[1]. Mahua biodiesel is produced from the seed oil of Mahua by a simlilar process of transestrification, in which seed oil is reacted with methanol and base catalyst at a specific temperature and time[2].

Mahua oil has high acidic value so it needs pretreatment process before transestrification process. Yield output is dependent upon the amount of alcohol, reaction temperature and reaction timings[3]. The alcohol used in this process is methanol as it is more reactive than the ethanol. The base catalyst used for production of biodiesel is KOH (Potassium Hydroxide) because it is also more reactive than the NaOH (Sodium Hydroxide).

Mahua biodiesel has better emissions characteristics than conventional diesel[4] but due to its high cloud and pour point temperatures, its cold flow characteristics are poor. To improve the cold flow characteristics of Mahua biodiesel, we need to improve its cloud and pour point. It can be achieved by some blending with diesel, kerosene, ethanol etc[5]. But as diesel and kerosene fuel has bad effects upon emissions characteristics so they can not be mixed. That leaves us with ethanol which has good effect on emissions as well as cold flow properties[6].

In this experimentation, two samples of Mahua biodisel were prepared with both have different parameters to study them like methanol to seed oil ratio, quantity of catalyst, reaction timings, reaction temperature etc. In order to improve its cold flow characteristics two more samples of Mahua biodiesel are made. Each has different proportions of ethanol added to it.

**Author for correspondence*

2. Material and Method

Raw Mahua oil was purchased from Himani International, Delhi and Methanol (99%) pure was purchased from Oswal Scientific Stores (Chandigarh). The base catalyst used for transestrification process was Potassium Hydroxide (KOH) in the pallet form. For pre-treatment process Sulphuric acid (H_2SO_4) was used as a catalyst. The experiments were performed in a laboratory scale apparatus, which contained 1 litter glass flask, stirring rod and a water bath whose maximum temperature was 100°C. The flask was kept inside the water bath and manually stirred the solution after 5 min interval.

2.1 Preheating

The Mahua seed oil was heated up to 100°C temperature in a water bath for 30 min time and stirred manually after 5 min of interval. This was done to remove water contained from seed oil because water‘s boiling point is 100°C and to remove unwanted dirt by filtering it.

2.2 Pre-treatment

The pre-treatment process was done to reduce the FFA (Free Fatty Acid) from 12% to less than 1%. In simple words; pre-treatment process was done to reduce the acidic value of raw Mahua seed oil.

Two samples were made, each containing different proportion of methanol to oil.

Sample 1 (MB100M50) contained preheated Mahua oil, 50% vol./vol. methanol to oil and 1% vol./vol. Sulphuric acid (H_2SO_4) as base catalyst. Reaction was carried for two hours in a glass beaker inside a water bath whose temperature was kept from 60°C to 80°C and solution was stirred manually after 5 min interval. Allow the mixture to settle for one hour inside a separating funnel.

$$R^1\,COOH + ROH = R^1\,COOR + H_2O$$

Pre-treatment Reaction

Sample 2 (MB100M75) contained pre-heated Mahua oil, 75% vol./vol. methanol to oil and 1.5% vol./vol. Sulphuric acid (H_2SO_4) as base catalyst. Reaction was carried for two hours in a glass beaker inside a water bath whose temperature was kept from 65°C to 90°C and solution was stirred manually after 5 min interval. Allow the mixture to settle for one hour inside a separating funnel. This Figure 1 shows the pre-treatment process.

Figure 1. Pre-treatment process.

2.3 Transestrification

Transestrification process was done to remove the glycerol from seed oil in order to get methyl ester solution from it. In this process, pre-treated seed oil was used and heat it up to 60°C temperature inside a water bath in a glass beaker.

For sample 1; Meth oxide solution was created which had 40% vol./vol. methanol and 1% wt./vol. Potassium Hydroxide (KOH) in pallet form. Mixed them in a separate flask with cork on it and shake this flask for at least 10 min.

Add Meth oxide solution to pre-treated Mahua oil which was kept at 60°C inside water bath and carried out this reaction for 30 min and temperature of water bath was varied from 60°C to 70°C, manually stirred the solution after 5 min of interval. And allow settling of solution for overnight inside a separating funnel.

For sample 2; Meth oxide solution was created which had 50% vol./vol. methanol and 1% wt./vol. Potassium Hydroxide (KOH) in pallet form. Mixed them in a separate flask with cork on it and shake this flask for at least 10 min.

Add Meth oxide solution to pre-treated Mahua oil which was kept at 60°C inside water bath and carried out this reaction for 60 min and temperature of water bath was varied from 60°C to 70°C, manually stirred the solution after 5 min of interval. And allow settling of solution for overnight inside a separating funnel. Figure 2 shows the transestrification process.

Figure 2. Transestrification process.

2.4 Separation

After overnight settling of solution inside separating funnel, solution made two layers: Lower layer was glycerine and upper layer was methyl ester. Remove the lower layer of glycerol carefully by opening the bottom end of separating funnel. Before doing this make sure the glass cork on the separating funnel was removed. And finally remove the upper layer of Methyl Ester of Mahua and place it in a separate beaker or bottle. Figures 3 and 4 shows the Mahua biodiesel placed for settling and after overnight settling time.

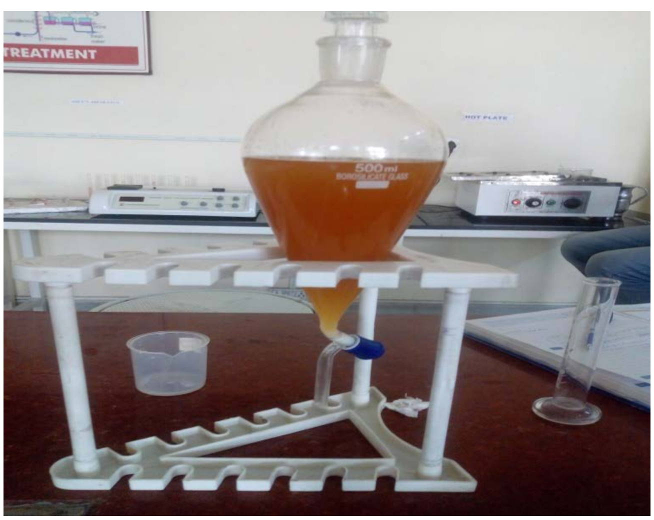

Figure 3. Mahua biodiesel placed for settling.

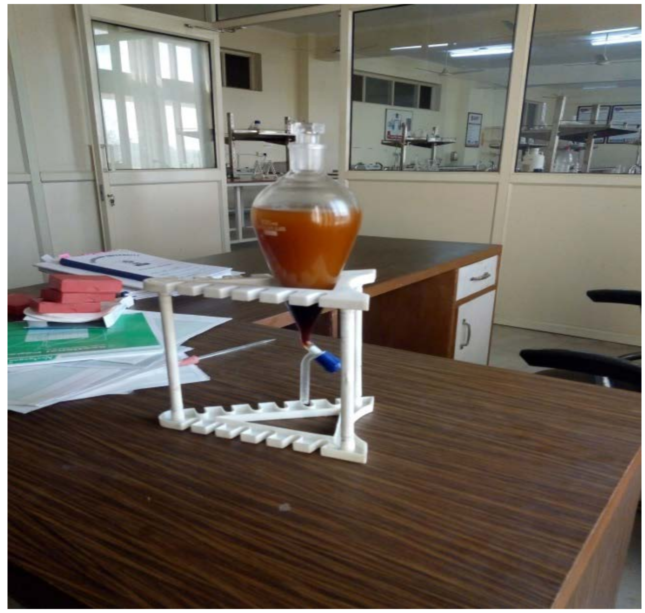

Figure 4. After overnight settling time.

2.5 Water Washing

After separating the methyl Ester layer from glycerine, due to cold weather some glycerine still present inside the methyl ester solution. In order to remove this unwanted glycerine from methyl ester solution, hot water washing was required.

In this process we normally added the hot water into the solution which was kept inside the separating funnel and remove the lower layer of glycerine after mixing the hot water to it. Keep repeating this process until we got a transparent layer of water at bottom of the separating funnel that means upper layer of methyl ester was free from glycerine. Now heat the final solution of methyl ester 100°C in order to remove any water contained present inside the methyl ester solution. This figure shows the water washing of Mahua biodiesel. Table 1 shows the comparison of physiochemical properties.

3. Improving Cold Flow Characteristics

Most of biodiesel properties are similar to fossil fuel based mineral diesel but improving the biodiesel cold temperature flowing characteristic is one of main challenges using it as an alternative fuel for C.I. engine[7].

Table 1. Comparison of physio-chemical properties

Sr.No	Property	Raw Mahua Oil	MB100M50	MB100M75	Mineral Diesel	ASTM (6751-02)
1	Density (kg/m^3)	950	882.7	875.3	830	850-879
2	Specific Gravity	0.95	0.883	0.875	.830	0.85-0.94
3	Kinematic Viscosity (cst)	24.99	3.99	3.57	2.60	1.9-6.0
4	Flash Point (^{o}C)	235	195	180	50	Min130
5	Fire Point (^{o}C)	245	205	190	60	Min135
6	Cloud Point (^{o}C)	12	8	6	-3	5-12
7	Pour Point (^{o}C)	8	3	2	-8	3-5

Biodiesel fuels are produced from vegetable oils, animal fats etc. which have high amount of saturated fat content which shows high cloud point and pour point[8].

The pour point occurs at low temperature than cloud point. Pour point is a temperature at which biodiesel become semi-solid and losses its flowing characteristics. And cloud point is a temperature at which biodiesel form a cloudy appearance[9].

3.1 Method

Pour point and cloud point tests show the cold temperature flow characteristics of biodiesel[10]. These were tested for the blends of methyl ester of Mahua (MME) with ethanol (10% and 20% sample) i.e. MB90E10 (MME 90% and ethanol 10%) and MB80E20 (MME 80% and ethanol 20%).

3.2 Cloud Point and Pour Point Experimental Setup

A sample of biodiesel was poured into glass tube and kept it inside the cloud point and pout point tester then place the cork with thermometer on it. Fill the surrounding of glass tube with cold water or ice in order to achieve desirable effect on biodiesel property like pour point and cloud point properties.

Note down the temperature at which biodiesel was formed cloudy appearance (i.e. cloud point) and temperature at which it formed semi solid state (i.e. pour point). Figure 6 shows the cloud and pour point setup. Table 2 shows the comparison of physio-chemical properties.

Figure 5. Water washing of Mahua biodiesel.

Table 2. Compression of physio-chemical properties

Sr. No.	Property	Raw Mahua Oil	MB100M75	Mineral Diesel	M90E10	M80E20	ASTM (6751-02)
1	Density (kg/m^3)	950	875.3	830	861.5	848.4	850-879
2	Specific Gravity	0.95	0.875	0.830	0.861	0.848	0.85-0.94
3	Kinematic Viscosity (cst)	24.99	3.57	2.60	3.10	2.65	1.9-6.0
4	Flash Point (^{o}C)	235	180	50	60	56	Min130
5	Fire Point (^{o}C)	245	190	60	68	63	Min135
6	Cloud Point (^{o}C)	12	6	-3	4	3	5-12
7	Pour Point (^{o}C)	8	2	-8	-1.5	-3.5	3-5

Figure 6. Cloud and pour point setup.

3.3 Results and Discussion

This Figure 7 shows the comparision of density.

Density is calculated at room temperature (20°C) and density of raw Mahua oil is found more than other samples. Figure 8 shows the comparision of kinematic viscosity.

Kinematic viscosity is calculated at 40°C teamparature by using Redwood Visco meter. It is found that MB100M75 has better viscosity than MB100M50 but not better than diesel. Figure 9 shows the comparision of flash point.

Flash point is calacuated with the help of Pensky Martins Close Cup and it shows that flash point of raw Mahua oil is more than other samples. Mahua biodiesl also have flash point more than diesel. Figure 10 shows the comparision of cloud point.

Cloud point is calculated in a cloud point and pour point tester. And it is found that Mahua biodiesel MB100M75 has lower cloud point than other samples of biodiesel but not less than diesel. Figure 11 shows the comparison of pour point.

Pour point is calculated in a cloud point and pour point tester. And it is found that Mahua biodiesel MB100M75 has lower cloud point than other samples of biodiesel but not less than diesel.

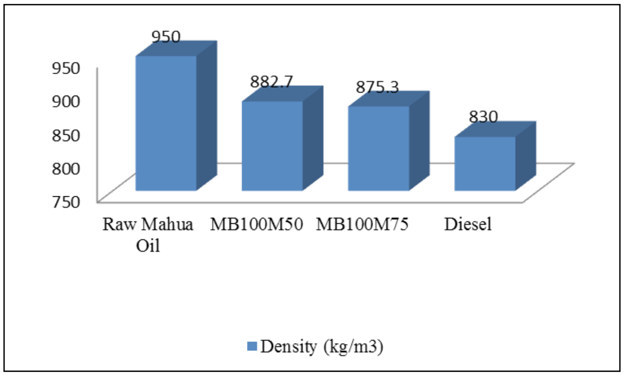

Figure 7. Comparision of density.

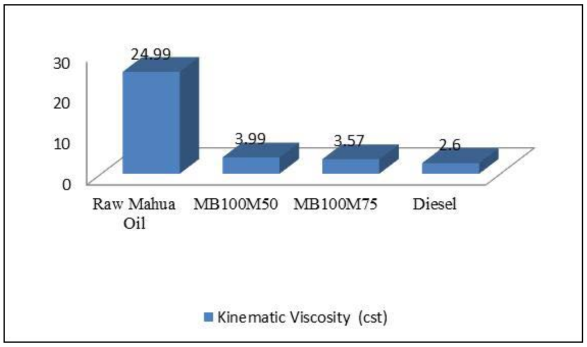

Figure 8. Comparison of kinematic viscosity.

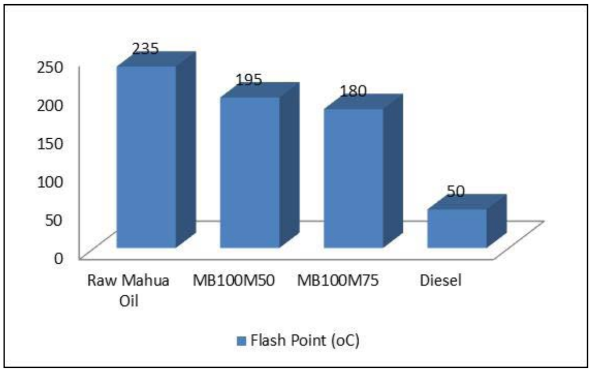

Figure 9. Comparison of flash point.

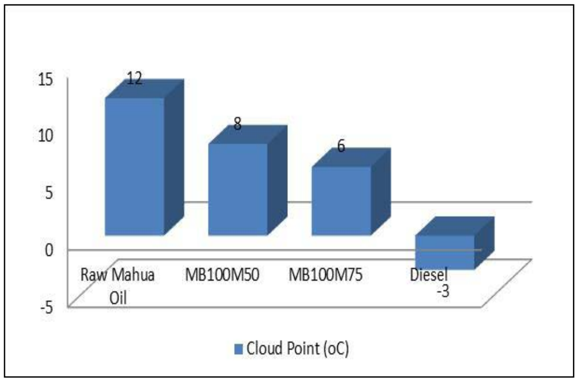

Figure 10. Comparison of cloud point.

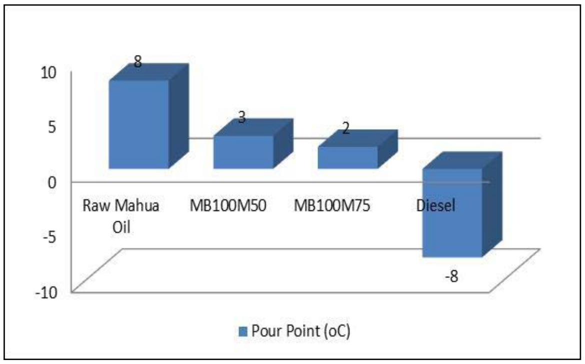

Figure 11. Comparison of pour point.

4.1 Cold Flow Properties Result

Figure 12 shows the comparision of cloud point of improved samples. It shows that the cloud point of MB80E20 (Mahua biodiesel 80% and ethanol 20%) is lower than other samples of biodiesel but higher than diesel. It means that MB80E20 sample has better cold flowing characteritics than other biodiesel samples but better than diesel.

Figure 13 shows the comparision of pour point of improved samples. It shows that the pour point of MB80E20 (Mahua biodiesel 80% and ethanol 20%) is lower than other samples of biodiesel but higher tha diesel. It means that MB80E20 sample have better cold flowing characteritics than other biodiesel samples but better than diesel.

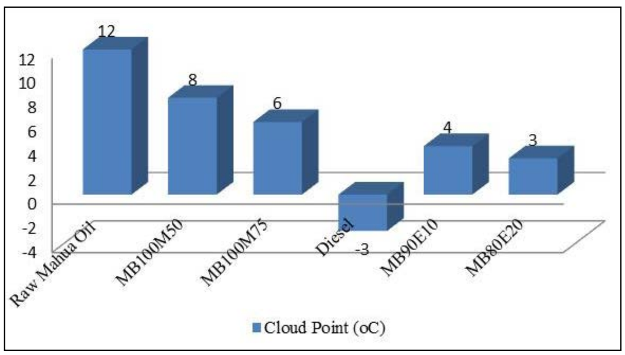

Figure 12. Comparing cloud point of improved samples.

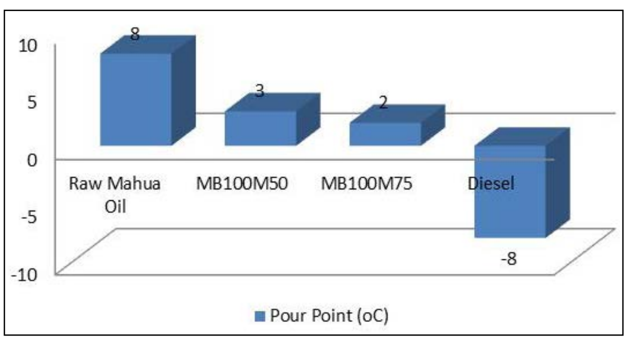

Figure 13. Comparing pour point of improved samples.

5. Conclusions

In this experimentation, the various results were found and are discussed as follows:

- Pre-treatment can be used to reduce the FFA value.
- MB100M75 has better physic-chemical properties than other biodiesel samples.
- Most physio-chemical properties were under the limits of ASTM standards.
- Addition of ethanol to biodiesel can improve the cold flow characteristics of biodiesels.
- MB80E20 sample had better pour and cloud point than other samples of biodiesel.

This work can be further extended by testing its combustions, performance and emissions characteristics on C.I. engine.

6. Acknowledgment

The authors acknowledge the assistance provided by Department of Automobile Engineering, Chandigarh University and technical support by Mr. Navdeep Sharma Dugala, Assistant Professor and Coordinator of Automobile Engineering Department, Chandigarh University,Gharuan (Punjab).

7. References

1. Meena R, Tak M, Nukani A. Biodiesel production from Mahua (Madhuca Indica) and its characterization through a TLC analysis. IOSR Journal of Mechanical and Civil Engineering (IOSR-JMCE); 2015. p. 77–83.
2. Guharaja S, Moorthy SD, Hasan IZ, Arun B, Ahamed JI, Azarudheen J. Biodiesel production from Mahua (Madhuca Indica). International Journal of Nano Corrosion Science and Engineering. 2016; 3(1):34–47.
3. Ghadge SV, Raheman H. Process optimization for biodiesel production from Mahua (Madhucaindica) oil using response surface methodology. Bioresource Technology. 2006; 97(3):379–84. PMid: 15908200. https://doi.org/10.1016/j.biortech.2005.03.014
4. Kumar A, Ram R, Tirkey JV, Shukla SK. Effect on performance and emission characteristics on direct injection diesel engine fueled with Mahua biodiesel. Conference paper Environmental Systems and Sustainable Development; 2016. 2.
5. Verma P, Sharma MP, Dwivedi G. Evaluation and enhancement of cold flow properties of palm oil and its biodiesel. Energy Reports. 2016; 2:8–13. https://doi.org/10.1016/j.egyr.2015.12.001
6. Bhale PV, Deshpande NV, Thombre SB. Improving the low temperature properties of biodiesel fuel. Renewable

Energy. 2009; 34(3):794–800. https://doi.org/10.1016/j.renene.2008.04.037

7. Rathore Y, Ramchandani D. Mahua biodiesel: An alternative to conventional diesel fuel. IOSR Journal of Mechanical and Civil Engineering (IOSR-JMCE). 2016; 16:1–10.
8. Kumar A, Veeresh Babu A, Tyagi CH. Production of Mahua methyl ester as a fuel replacement to diesel (a laboratory study). International Journal of Engineering and Innovative Technology (IJEIT). 2015; 5(6):1–4.
9. Lopes PM, Muller D, Harrison R, Bordado JC. Effect of additives to improve the performance of biodiesel at low temperatures. Arizona Chemical/Instituto Superior Tecnico. 2007. 1–7. PMid: 17224990.
10. Shrestha DS, Van Gerpen J, Thompson J. Effectiveness of cold flow additives on various biodiesels, diesel and their blends. American Society of Agricultural and Biological Engineers. 2008; 51(4):1365–70. https://doi.org/10.13031/2013.25219