International Journal of Energy and Power Engineering
2015; 4(5-1): 48-53
Published online August 31, 2015 (http://www.sciencepublishinggroup.com/j/ijepe)
doi: 10.11648/j.ijepe.s.2015040501.18
ISSN: 2326-957X (Print); ISSN: 2326-960X (Online)

Development of Transesterification System with Acid and Base Homogeneous Catalysts For Mangifera Indica Seed Oil to Mangifera Indica Methyl Ester (MOME Biodiesel)

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To cite this article:
Shubhangi S. Nigade, Sangram D. Jadhav, Abhimanyu K. Chandgude. Development of Transesterification System with Acid and Base Homogeneous Catalysts For Mangifera Indica Seed Oil to Mangifera Indica Methyl Ester (MOME Biodiesel). *International Journal of Energy and Power Engineering*. Special Issue: Energy Systems and Developments. Vol. 4, No. 5-1, 2015, pp. 48-53.
doi: 10.11648/j.ijepe.s.2015040501.18

Abstract: The depletion of resources, increased cost of fossil fuel and increased environmental awareness reaching the critical condition. Development of viable alternative fuels from renewable resources is gaining the international attention and acceptance. The vegetable oils have the potential of alternative fuel for compression ignition engines by converting it into biodiesel. The mangifera indica oil is a nonedible vegetable oil, available in large quantities in mangifera indica cultivating countries including India. Very little research has been done on utilization of oil in general and optimization of transesterification process for biodiesel production. However, direct base catalyzed transesterification produced no biodiesel due to the high Free Fatty Acid (FFA) value of the oil. Hence, acid pretreatment was preferred prior to base transesterification which afforded a significant reduction of the FFA value from 3.3% to 0.9% . Various input parameters like oil-to-methanol molar ratio (1:08, 1:12 and 1:16), catalyst type (NaOH, KOH and NaOCH3), catalyst concentration (0.5, 1 and 1.5 wt %) and reaction temperature (59, 64 and 69°C) were studied. The optimum conditions for transesterification process are: 1:12 oil-to-methanol molar ratio, 1.0 wt.% catalyst concentration, KOH catalyst, & 64°C reaction temperature. The optimum yield of MOME was 89.8%. The biodiesel produced (MOME) is within the limits prescribed by EN-14214 standard.

Keywords: Biodiesel, Extraction, Mangifera Indica, Pretreatment, Transesterification

1. Introduction

India currently ranks as the worlds 11th greatest energy producer accounting for about 2.4% of the world's total annual energy production, while it ranks as the 6th largest energy consumer accounting for about 3.3 % of the world's total annual energy consumption. Currently India is such a country where present level of energy consumption by world standard is very low, with per capita energy consumption is less than 500 Kgoe (Kilogram oil equivalent) compared to Global average of nearly 1800 Kgoe. The major part of all energy consumed in most parts of the world comes from fossil sources such as petroleum, coal and natural gas. However, these non-renewable sources will be exhausted in near future. Recent assessments of remaining petroleum reserves show the world will soon face a relentless oil-supply conventional crude oil is projected to peak and decline irreversibly. Alternative sources for petroleum products will then be critical. In India 95 % energy need of transportation sector are provided by the Diesel and the demand for diesel is five times higher than the diesel it was estimated that for sustaining India's 8% average annual economic growth and to support its growing population. India needs to generate 2 to 3 fold more energy than present It is estimated that India has only 0.4% of the world's proven reserves of crude oil .India meets about 70 % of its petroleum requirement through import which are expected to expand in the coming years. In India volume of crude oil imported increased 14 fold from 11.66 million tons during 1970-71 to 163.59 million tons by 2010 to 2011.During last 7 years; India's foreign exchange outflow due to this purpose has increased 5 fold because of escalation of international oil prices [1-2].

If the nation's source of petroleum products continues to be limited to conventional crude oil, this situation is certain to become worse. Widely acknowledged estimates of remaining recoverable reserves of conventional crude oil worldwide total 1 trillion barrels. Thus, the search for alternative sources of renewable and sustainable energy has gained importance with the potential to solve many current social issues such as the rising price of petroleum crude and environmental concerns like air pollution and global warming caused by combustion of fossil fuels [3-4]. The term biofuel or renewable fuel is referred to as solid, liquid or gaseous fuels that are predominantly produced from biomass. Liquid and gaseous biofuels have become more attractive recently because of its environmental benefits. Biofuels are non-polluting, locally available, accessible, sustainable and reliable fuel obtained from renewable sources [5-6]. Among other reasons why biofuels are considered as relevant technologies by both developing and industrialized countries are: energy security, environmental concerns, foreign exchange savings, and socio-economic issues related to the rural sectors of all countries in the world [7]. In recent years, many studies have investigated the economic and environmental impacts of biofuels, especially bioethanol, biodiesel, biogas, and biohydrogen [6]. Biodiesel (fatty-acid alkyl esters) is a renewable and environmentally friendly energy source. It can be produced from plant oils and animal fats. Several techniques are available for biodiesel production. The most commonly used technique is transesterification in which triglycerides are reacted with alcohol, usually methanol, in the presence of a catalyst, usually potassium or sodium hydroxide (KOH or NaOH), to produce mono alkyl esters. Many factors affect the biodiesel yield and process economics. The most important factors are alcohol type, alcohol/oil molar ratio, reaction temperature and time, catalyst type and amount and water content of the reactants [8]. Mangifera indicas belong to the genus Mangifera of the family Anacardiaceae. The genus Mangifera contains several species that bear edible fruit. Most of the fruit trees that are commonly known as mangifera indicas belong to the species Mangifera indica. The other edible Mangifera species generally have lower quality fruit and are commonly referred to as wild mangifera indicas. Mangifera indica fruit is classed as a drupe (fleshy with a single seed enclosed in a leathery endocarp).

Fruits from different varieties can be highly variable in shape, color, taste and flesh texture. Fruit shapes vary from round to ovate to oblong and long with variable lateral compression. Fruits can weigh from less than 50 g (0.35 lb) to over 2 kg (4.4 lb). The fruit has a dark green background color when developing on the tree that turns lighter green to yellow as it ripens [9]. Currently, Nigerian government has shown great interest in Jatropha and other biofuel plants. The aim of the government is to gradually reduce the nation's dependency on gasoline, reduce environmental pollutions as well as create commercially viable industry that can precipitate domestic job [10-11,26-30].

India is the largest producer of mangifera indica in the world, with the annual production about 15.19 million tonnes. Indian share in production of mangifera indica is 42.04% of world production. Mangifera indica is by-product of mangifera indica, which contain approximately 25.6 to 32.6% oil("UN FAOSTAT 2012.pdf", S. S. Raju et al. 2012, Thammarat 2013). Such a huge amount of feedstock volume, high oil contents & low cost favors mangifera indica oil for biodiesel production in India (Tapasvi et al. 2005). A large green tree mainly valued for its fruits, raw and ripe. It can grow up to 15-30 m tall and its yield in kg per tree is given in table no. 1.

Table 1. *Yield of Mangifera indica as per age.*

Sr. No	Age (Years)	Fruit no./ tree	Yield (kg/yr/tree)
1	5-8	450	292
2	9-10	800	657
3	11-25	1250	892

In view of this, it was decided to optimize the transesterification process for production of biodiesel using homogeneous catalysts. The main objective of research was to maximize yield with respect to input variables; methanol to oil molar ratio, catalyst type, catalyst amount and reaction temperature.

2. Experimentation

2.1. Materials

Methanol (Purity 99.8%, IR spectrum), potassium hydroxide pellets (Purity 98.0%), sodium hydroxide pellets (Purity 98.0%), N-hexane (Purity 99.0%, IR spectrum), Acetone hexane (Purity 99.0%, IR spectrum), Silicon oil (oil bath upto 250°C), Sulfuric acid (Purity 98.0%), Stearic acid (Purity 98.0%), Palmitic acid (Purity 98.0%), Oleic acid, Linoleic acid(Purity 90.0%), Linolenic acid (Purity 98.0%), Arachidic acid (Purity 90.0%), 1250 Grade1 filter paper (10-13µm, <0.06 Cenizas), phenolphthalein pH indicator were purchased Thomas baker Chemicals pvt. Ltd. India. The kernels of Mangifera indica were collected at canning industries around coastal area konkan, India.

2.2. Extraction of the Oil

Before the extraction process, mangifera indica kernels were dried overnight at 58°C in an oven to remove the excess moisture. The dried seeds were then weighted and crushed into fine particles of 0.5 to 10 mm in size. The oil was then extracted using Soxhelet extractor with N-hexane as the solvent. The duration for each batch of extraction was fixed at 4h; while the volume of solvent per kilogram of seed was varied from 4 liter to 6 liter for maximization of oil yield. The oil was recovered at the RBF which put into the heating mental at the bottom. The spiral coil condenser is used for recovery of solvent which get collected in RBF and re-used again in the process till completion of extraction. The extracted oil was then measured to calculate the content of oil in the kernel of mangifera indica. The physiochemical properties and fatty acid composition of mangifera indica oil (MIO) are shown in Table 2 and 3.

Table 2. *Physiochemical properties of mangifera indica oil.*

Sr. No	Physical character	Value
1	Refractive Index at 40°C	1.4560
2	Iodine Value	47.3
3	Saponification value	192.4
4	Unsaponificable matter	1.2%
5	Specific gravity	0.998
6	colour	Dark Yellow

Table 3. *Fatty acid composition of the mangifera indica oil.*

Fatty acid	Chemical Structure	Percentage (%)
Palmitic	$C_{16}H_{32}O_2$	5.6
Stearic	$C_{18}H_{36}O_2$	40.3
Oleic	$C_{18}H_{34}O_2$	46.6
Linoleic	$C_{18}H_{32}O_2$	5.1
Arachidic	$C_{20}H_{40}O_2$	3.2
Linolenic	$C_{18}H36O_2$	0.3

2.3. Acid Pretreatment Process

The pretreatment was conducted in a corked 250ml flat bottom flask, placed on a hot plate magnetic stirrer preset at the required temperature. In the experiments, flasks loaded with Mangifera indica oil samples was first heated to the designated temperature of 500C[17, 24, 29-31]. This was followed by the addition of the methanol and sulfuric acid (The solution of concentrated H_2SO_4 acid 1.0% based on the weight of oil, and the oil to methanol ratio of 1:6 by volume and a reaction time of 70min). The Transesterification pretreated products oil was separated in a tap funnel to obtain the upper layer, which was then washed with water several times until the pH of the washing water was close to 7.0 [14]. The resultant pretreated oil was dried in an oven before the subsequent transesterification process [18, 26-30].

2.4. Transesterification

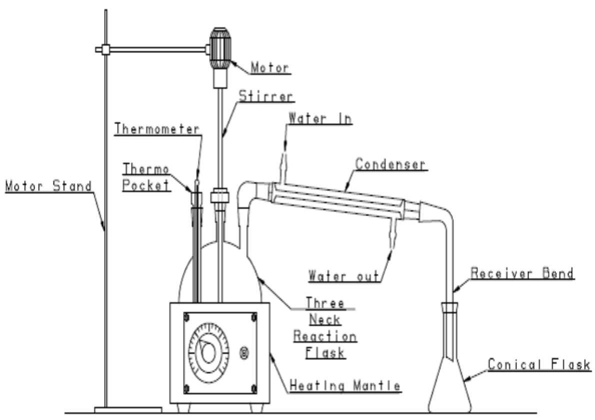

Fig. 1. *Experimental setup for Pretreatment and Transesterification.*

Base catalyzed transesterification was carried out according to the Ireland method 1. In a 250 ml conical flask equipped with a magnetic stirrer. 30ml of the extracted oil was taken in flask and potassium hydroxide (1 percent of oil's weight) dissolved in methanol (22.5 percent of oil's weight) was added to flask (as shown figure 1). Stirring was continued for 90mins at 600C, the mixture was transferred to a separatory funnel and glycerol was allowed to separate, leaving the upper layer biodiesel and the lower layer glycerin [19, 30-31].

2.5. Experimental Conditions

Experiments were planned to ascertain the oil/methanol ratio (w/w), catalyst concentration, reaction temperature and agitation intensity on transesterification reaction. The ratio of Oil/Methanol was varied as per w/w 4:1, 5:1, 6:1, the catalysts was sodium hydroxide and its concentrations were varied as 0.25, 0.50, 0.75, 1.00 and 1.50% of the oil. Reaction temperatures considered were 50, 55, 60 and 65°C and the agitation intensity were varied through 150, 300, 450, 600 and 700 rpm.

2.6. Biodiesel Separation and Washing

After obtaining the biodiesel phase, methyl ester was washed with hot water three times until the residual catalyst is finally off the solution. Warm water at temperature of about 500C, usually ratio 1:1 to the biodiesel was used in each washing step to clean up the esters. Finally, the biodiesel was dried in an oven at 105 degree for 30mins [19-23,30].

2.7. Fuel Properties

The following properties of the biodiesel produced were determined: density and specific gravity [11], kinematic viscosity 40°C (ASTM D 445), flash point (ASTM D 93), Sulfated Ash (ASTM D847) carbon residue (ASTM D524).

3. Results and Discussion

3.1. Effect of Molar Ratio

In stoichiometric transesterification reaction, each mol of triglyceride requires three moles of alcohol to produce three moles of fatty acid alkyl ester and one mole of glycerol, whereas esterification requires one mole of FFA and one mole of alcohol to generate one mole of ester and one mole of water. Since these reactions are reversible, excess alcohol is required to drive the reaction toward the product side for increasing and completing the conversion [22-27].

The average molecular weight of mangifera indica oil was calculated from its composition and accordingly the amount of methanol was taken in the reaction. The methanol to oil molar ratio varied from 1:8 to 1:16 to study its effect on yield of conversion process. The mean yield of MOME at different molar ratio 1:8, 1:12 and 1:16 are shown in Fig. 2. The effect of methanol in the range of 1:8 to 1:16 (molar ratio) was investigated. It was found that the ester yield increases with increase in molar ratio of methanol to vegetable oil. Molar ratio is in between 1:08 to 1:12 shows faster conversion rate compared to molar ratio after 1:12.

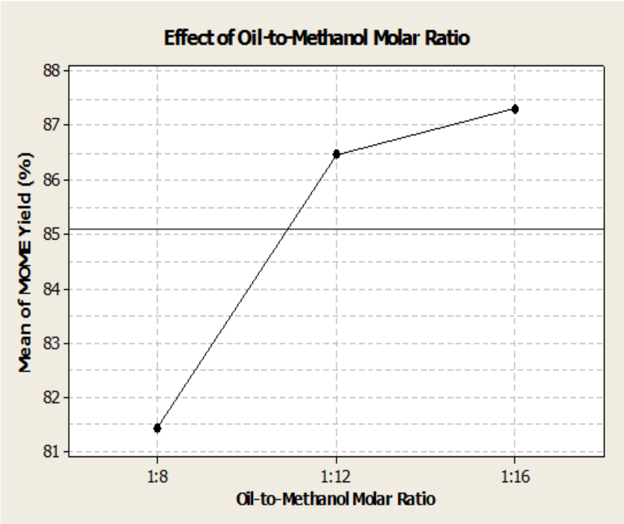

Fig. 2. *Effect of Oil-to-Methanol Molar Ratio.*

At low molar ratio, low proportion of methanol reduces the probability of braking bonds between glycerol and tryglyceriods reducing the yield. Though the stoichiometric molar ratio is 3:1, the general trend is increase in yield of reaction with molar ratio. Therefore molar ratio at 1:12 shows the optimum molar ratio for the yield of mangifera indica oil. However, when the ratio of oil to alcohol is too high, it could give adverse effect on the yield of fatty acid alkyl esters eg. phase separation of ester and glycerol, mass transfer problem between triglycerides etc.

3.2. Effect of Catalyst Type

The type of catalyst required in the transesterification process usually depends on the quality of the feedstock and method applied for the transesterification process. For a purified feedstock, any type of catalyst could be used for the transesterification process.

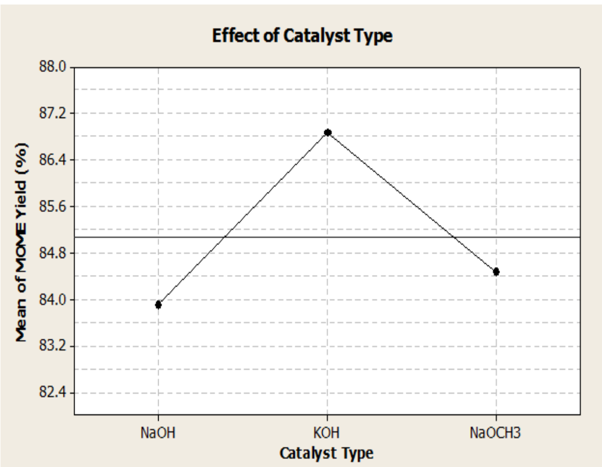

Fig. 3. *Effect of Catalyst Type.*

However, for feedstock with high moisture and free fatty acids contents, homogenous transesterification process is unsuitable due to high possibility of saponification process instead of transesterification process to occur. Two step transesterification processes had been suggested by several researchers (13-18,21). At present, homogeneous catalysts such as NaOH and KOH are primarily used by biodiesel industry due to their simple usage and short time required for conversion of oils to ester. The homogeneous catalysts (e.g NaOH, KOH and $NaOCH_3$) catalyst forms sodium and potassium methoxide due to its solubility in methanol which augment the completion of reaction. The main advantage homogenous acid and alkali catalysts are high yield and low cost. Transesterification process was carried out by using three homogeneous catalysts (NaOH, KOH, $NaOCH_3$). The results obtained are presented in Fig. 3. The output values represented in figure are mean percentage values of yield. There is significant effect of type of catalyst on yield. Amongst the homogeneous catalysts the output with KOH (86.6%) catalyst is greater than NaOH, $NaOCH_3$ catalyst.

3.3. Effect of Catalyst Concentration

Tests were conducted to study the effect of amount of catalyst used (concentration) on conversion of mangifera indica oil to ester. The results of the tests are presented in Fig. 4. The amount of catalyst used during the tests was varied from 0.5 to 1.5 % of weight of the oil in a step of 0.5. The yield of MOME increases with increase of catalyst concentration. If we see the rate of yield conversion it is faster upto 1.0 wt.% catalyst concentration after it there is slightly lower the yield conversion rate. As increase in catalyst concentration no doubt yield increases, this is due to availability of more active sites by additions of larger amount of catalyst in the transesterification process.

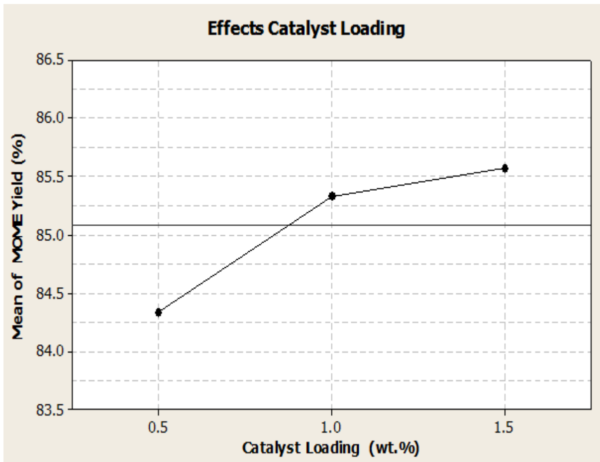

Fig. 4. *Effect of Catalyst Loading.*

However, on economic perspective, larger amount of catalyst may not be profitable due to cost of the catalysts itself. Hence for economic and performance points of view, 1.0 wt.% catalyst loading is optimum for mangifera indica oil.

3.4. Effect of Reaction Temperature

The reaction temperature was varied from 59 to 69 °C. The results of the test are presented in Fig. 5. The yield of mangifera indica oil increases with increase in temperature upto 64 °C and it drops beyond this value of temperature. The maximum value of mean yield (85.82%) was observed at 64 °C. It is interesting to note that maximum yield was observed at temperature 64 °C which is very close to boiling point of methanol (64.6 °C). The formation of vapour causes the volatilization and the momentum between monoglycerid, diglyceride, triglyceride and glycerol which helps for breaking the bond.

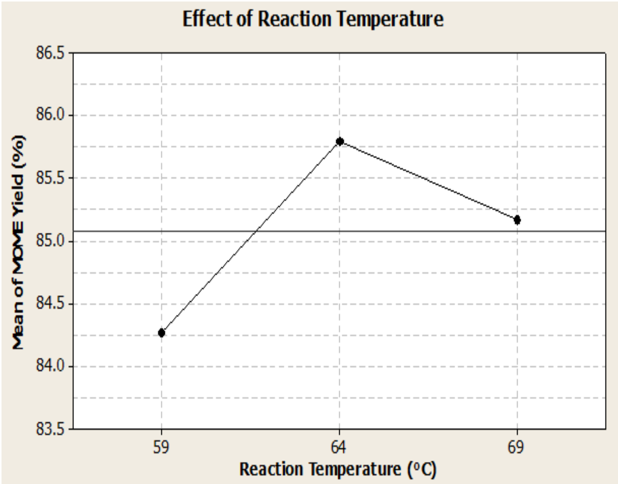

Fig. 5. *Effect of reaction Temperature.*

3.5. Biodiesel Characterization

The fuel properties of the biodiesel (Mangifera indica methyl ester) and mineral Diesel were determined using standard test procedures (are given in Table 4). The calorific value is a measure of the energy content of the fuel and is a very important property of biodiesel, which determines its suitability as an alternative to mineral Diesel. The calorific value of Mangifera indica methyl ester (MOME) and Mangifera indica oil is 43.1 and 42.3 MJ/kg, which is almost 96% and 93% of the diesel calorific value (44.8 MJ/kg), respectively.

Table 4. *Properties of diesel, mangifera indica oil and mangifera indica methyl ester (MOME).*

Property	Std.	Diesel	MVO	MOME
Specific gravity	---	0.839	0.912	0.872
Kinematic viscosity @ 40°C (cSt)	ASTM D445	3.18	38.46	4.62
Cloud Point (°C)	ASTM D2500	6	11	8
Pour Point(°C)	ASTM D2500	-7	1	-2
Flash Point(°C)	ASTM D93	68	310	176
Fire point(°C)	ASTM D93	103	332	179
Carbon Residue (%, w/w)	ASTM D189	0.1	0.8	0.38
Calorific Value (MJ/kg)	ASTM D240	44.8	42.3	43.1

The lower calorific value of MOME is because of the presence of oxygen in the molecular structure, which is confirmed by elemental analysis also. The flash point and fire point were tested with a closed cup Pensky Marten's apparatus. The flash point is the measure of the tendency of a substance to form flammable mixtures when exposed to air. This parameter is considered in the handling, storage and safety of fuels. The high value of flash point and fire point in the case of MOME represents it is a safer fuel to handle.

4. Conclusions

The present study was aimed to optimize transesterification process for mangifera indica oil using different types of homogeneous catalysts. The study involved following processes:

- Extraction of mangifera indica oil,
- Transesterification with different catalysts
- Catalyst removal from biodiesel
- Measurement of properties and quality of ester as product of the process.

Extensive work has been done mainly on jatropha and karanja oil. This may be partly due to a) the policy of Indian government to promote use of nonedible feedstock for alternative fuel b) large scale potential for their growth. Varity of crops are grown in different regions of the country. Hence regional feedstocks like mahua, simaruba, mangifera indica, neem, kokam, mahua etc., apart from jatropha and karanja, are also promoted for biodiesel production. Literature review on mangifera indica oil revealed that very little work has been done on this oil. This was one of the motivating factors to carry out the present study on mangifera indica oil. In west coast of India huge quantity (15.19 million of tonnes) of mangifera indica kernel is available. Hence there is good opportunity for obtaining biodiesel from this oil in India. High oil content, large availability, low cost of feedstock, huge amount of mangifera indica kernel per tree per year, low initial investment are the key features in favors of its use for biodiesel production.

Alkali catalyzed transesterification was studied with NaOH, KOH, and NaOCH3 as catalyst for biodiesel production from Mangifera indica oil. Apart from optimization of transesterification process, catalyst removal from biodiesel also has been studied in detail. Transesterification reaction parameters decide the yield of the ester, while catalyst removal decides the quality of ester.

5. Overall Conclusion

The effect of the parameters, namely molar ratio of oil-to-alcohol, catalyst type, catalyst concentration and reaction temperature on the yield of ester (biodiesel) was investigated. The optimum values of input parameters for maximum yield of biodiesel (MOME) were obtained as: 1:12 oil-to-alcohol molar ratio, KOH catalyst, 1.0 wt.% catalysts concentration and 64°C reaction temperature. The biodiesel produced is within the limits prescribed by ASTM standard. The results of the study revealed that KOH catalyst (homogeneous) is best for mangifera indica oil, which is one of the promising resources for biodiesel production and possible substitute for diesel.

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