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Optimization of Cotton Seed Methyl Ester and Mustard Methyl Ester from Transesterification Process

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Abstract

The most commonly used method for biodiesel preparation is via transesterification of vegetable oil using alkaline catalysts. Biodiesel yield and oil conversion are affected by operating conditions including the catalyst formulation and concentration. Application of alkaline catalysts can also lead to undesired soap formation. This study evaluated the alkaline catalyst effects on biodiesel yield and soap formation in transesterifying methanol with waste cotton seed oil and waste mustard oil at different catalyst concentrations. It was observed that methoxide catalysts led to better biodiesel yields. The methoxide catalysts not only accelerated the reaction but also elevated the conversion equilibrium. Based on experimental optimization, the operating conditions for maximizing cotton seed and mustard biodiesel yield and minimizing soap formation were potassium methoxide as catalyst at 1.5% (1.365 gm) for cotton seed biodiesel and 1.5% (1.305 gm) for mustard biodiesel, reaction temperature of 600C, and methanol-to-oil molar ratio of 5:1 for both the oils. Experimental verification gave 78.9% biodiesel yield from waste cotton seed oil and 87.9% biodiesel yield from waste mustard oil.

Keywords: Biodiesel, Alkaline catalyst, Transesterification, Waste cotton seed oil, Waste mustard oil.

Introduction

Several ways have been researched and demonstrated for preparing biodiesel from waste vegetable oils, but base-catalysed transesterification is still the most widely used method in biodiesel production.

The most commonly used alkaline catalysts in the biodiesel industry are potassium hydroxide (KOH) which is inexpensive, easy to handle in transportation and storage, and are preferred by small producers. Alkyl oxide solutions of sodium meth oxide ($NaOCH_3$) or potassium meth oxide ($KOCH_3$) in methanol, which are now commercially available, are the preferred catalysts for large continuous-flow production processes. In transesterification, the effective species of catalysis is the meth oxide radicals (CH3O−). The activity of a catalyst depends upon the amount of meth oxide radicals available for the reaction. For sodium or potassium hydroxide, meth oxide ion is prepared by reacting hydroxide and methanol:

$$OH + CH_3OH \longrightarrow CH_3O + H_2O \quad (1)$$

This reaction also yields water that remains in the system. Hydrolysis of triglycerides and alkyl esters may occur due to the presence of water, which further leads to the formation of
free fatty acids and thus to undesired soap. Saponification will also occur if a strong base, e.g., NaOH or KOH in the system by reacting with esters and triglycerides directly. On the other hand, the water problem can be avoided if sodium and potassium meth oxide solutions, which can be prepared water-free, are applied.

The extent of transesterification and side reactions depends upon the types of feedstock, catalyst formulation, catalyst concentration, reaction temperature, and methanol to oil ratio. Free fatty acid and moisture content in the reactant mixture also play important roles in biodiesel production .In the transesterification of vegetables oils and animal fats, each mole of triglycerides reacts stoichiometrically with 3 moles of a primary alcohol and yields 3 moles of alkyl esters (biodiesel) and 1 mole of glycerol (by-product). The actual mechanism of the transesterification reaction consists of sets of equilibrium reactions in series and all of the reactions are reversible.

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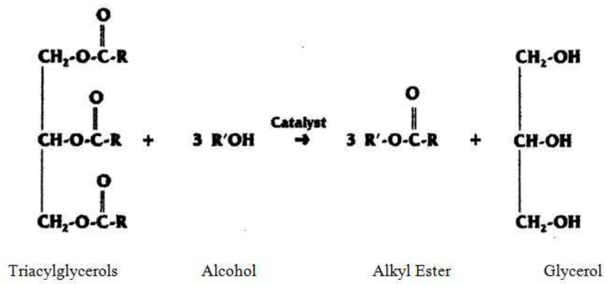

Figure 1

Where R is a long hydrocarbon chain, sometimes called fatty acid chains.

Materials and Methods

Materials

The waste cotton seed and waste mustard oil used in this study was collected from a local snacks shop in Patiala, Punjab, India. The biodiesel from the both the oils was extracted at Mechanical Engineering Research and Development Organization, Ludhiana. Water bath shaker is used for transesterification of waste cotton seed oil and waste mustard oil. The effect of process parameters shown in Table 1 were studied to standardize the transesterification process for estimating recovery of ester as well as recovering ester of lowest possible viscosity. In order to standardize the process parameters, four levels of catalyst (KOH) concentration (1%, 1.5% 2%, and 2.5%) were set. The transesterification was done at 5:1 molar ratio and then allowed to settle for 1 hour in order to obtain maximum recovery of ester with lowest possible kinematic viscosity. Total 8 ester samples were prepared to study the effect of the four levels of catalyst concentration on ester recovery and subsequent measure of their kinematic viscosity.

Table 1 Process parameters selected for standardization of transesterification process (waste cotton seed oil and waste mustard oil)

Optimizing parameter	Level selected
Preheating Temperature (^{0}C)	60^{o}C
Molar Ratio	5:1
Catalyst concentration	1%, 1.5%, 2%, 2.5%
Reaction time (Minutes)	60 minutes
Reaction Temperature (^{0}C)	60^{0}C
Settling Time	1 hr

Results and Discussions

Effect of catalyst on the yield of biodiesel: - The effect of KOH concentration was studied in the range of 1-2.5% (weight of KOH/weight of oil).The reaction temperature was kept constant at 60^0C. It was found that the ester recovery increases as the amount of catalyst decreased from 2.5-1%. Ester recovery decreases drastically as KOH concentration increases above 2.5% whereas ester recovery increase as KOH concentration reduces to almost 1%. This lesser recovery at high KOH concentration may possibly be due to high soap formation. It also shows that the lowest molar ratio of alcohol to oil, lesser amount of KOH can be used.

The effect of composition of KOH on the yield of biodiesel prepared from waste cotton seed oil is shown in below Table 2

Yield of Biodiesel obtained from waste cotton seed oil

Amount of oil (gms)	conc. of KOH	Methanol (gms)	Amount of Yield (gms)
91	1% (0.91 g)	18.2	77.12
91	1.5% (1.365 g)	18.2	71.82
91	2% (1.82 g)	18.2	64.18
91	2.5% (2.275 g)	18.2	53.94

The effect of composition of KOH on the yield of biodiesel prepared from waste mustard oil is shown in below Table 3

Yield of Biodiesel obtained from waste mustard oil

Amount of oil (gms)	Conc. Of KOH	Methanol (gms)	Amount of yield (gms)
87	1% (0.87 g)	17.4	83.23
87	1.5% (1.305 g)	17.4	76.49
87	2% (1.74 g)	17.4	65.54
87	2.5% (2.175 g)	17.4	58.82

Effect of catalyst on the properties of biodiesel: - The varying concentration of KOH affects the yield of biodiesel. When the biodiesel was prepared from oil by the transesterification process, generally two properties were checked. These properties were FFA Value (Free Fatty Acid) and Viscosity. Viscosity is an important property of a bio diesel. With extremely low viscosities may not provide sufficient lubrication for the pumps and Injector plungers. They can promote abnormal wear and cause injector and injector pump leakage. Higher viscosity is also not desirable as too viscous fuel increases pumping losses in injector pump and injectors, which reduces injection pressure resulting in poor atomization and inefficient mixing with air ultimately affecting the combustion process. So the viscosity is an important property to check because it was neither to low nor too high.

The second property was FFA Value. If the oil has a high water or free fatty acid (FFA) content the reaction will be unsuccessful due to saponification (saponification is defined as the reaction of an ester with a metallic base and water) commonly known as making soap, and make separation of the glycerol difficult at the end of the reaction. The FFA content of the raw oil will determine the quantity of biodiesel as the final product. A very low content of FFA (<0.2) can give a full 100% yield [1]. So these two properties were important to check when biodiesel is extract from oil.

The effect of KOH concentration on FFA value and Viscosity of cotton seed biodiesel is shown in below Table

Properties of cotton seed biodiesel

Amount of yield (gms)	conc. of KOH	FFA value	Viscosity
77.12	1%	0.141	4.2
71.82	1.5%	0.112	3.6
64.1	2%	0.090	3
53.94	2.5%	0.0564	2.5

The effect of KOH concentration on FFA value and Viscosity of mustard oil biodiesel is shown in below Table 5

Properties of mustard biodiesel

Amount of yield (gms)	Conc. Of KOH	FFA Value	Viscosity
83.23	1%	0.129	4.9
76.49	1.5%	0.0126	3.7
65.54	2%	0.0705	3.2
58.82	2.5%	0.0282	2.5

Conclusions

The recovery of esters by transesterification of waste cotton seed oil and waste mustard oil with methanol are affected by varying the composition of catalyst.

The recovery of cotton seed methyl ester at lowest kinematic viscosity (3.6 cSt) was 71.82 gram from 91 gram is possible at the following standardized concentration of catalyst.

a) Molar ratio: - 5:1
b) Reaction time: - 60 minutes
c) Catalyst concentration:- 1.5%
d) Reaction temperature:-60^{0}C
e) Settling time: - 1 hour

The recovery of mustard methyl ester at lowest kinematic viscosity (3.7 cSt) was 76.49 gram from 87 gram is possible at the following standardized concentration of catalyst.

a) Molar ratio: - 5:1
b) Reaction time:-60 minutes
c) Catalyst concentration: - 1.5%
d) Reaction temperature: - 60^{0}C
e) Settling time: - 1 hour.

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