

TRANSESTERIFICATION OF *SESBANIA BISPINOSA* SEEDS OIL FOR THE PREPARATION OF BIOFUEL

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ABSTRACT

Biodiesel was prepared from the non-edible oil extracted from the seeds of *Sesbania bispinosa* via transesterification. Different factors were studied to find the maximum yield of the biodiesel. It was found that 1:6 oil to methanol molar ratio, 1 %wt of catalyst concentration and 1h of heating at 60 °C with constant stirring produced maximum yield of biodiesel as 86%. Prepared biodiesel was characterized for different physico-chemical properties examining flash point (177 °C), density (0.870 g/cm³), kinematic viscosity (4.76 cSt), conradson carbon residue (0.0170 % wt), ash content (nill), distillation range (200-

329°C) sulphur content (0.014 % wt), calorific value (17256.60 btu/lb) and acid number (0.20 mg KOH/g). All the values were within the limits set by ASTM D 6751 standards.

KEYWORDS: Biodiesel, *Sesbania bispinosa*, physicochemical properties, kinematic viscosity.

INTRODUCTION

World peace is continuously threatened due to the depletion of non-renewable energy sources from the world and environment pollution due to the exhaust emissions of diesel engines.

Energy crisis, though not taken much seriously, can be the leading cause of the many problems in future. The use of a biodegradable, renewable and less toxic biodiesel could minimize all these problems. Biodiesel, major type of liquid biofuel, is alkyl esters of long chain of fatty acids derived from animal fat or plant oil. It is the best substitute for variety of diesel engines in place of diesel fuel. Biodiesel can be utilized easily in existing diesel engines with very little transformations in the form of neat fuel (B100) or its blends with petroleum based diesel. Engine lubrication improved and life extends due to the use of biodiesel. Moreover petroleum fuels releases a huge amount of hazardous chemicals in the environment like carbon dioxide, particulate matter and hydrocarbons that are carcinogenic and a major cause of global heating. The use of biodiesel can reduce the emission of these harmful gases to a large extent.

Edible plant based lipids comprises the large portion of biodiesel produced worldwide. Around 80% of biofuel in the European Union is produced from the oil extracted from of rapeseed and sunflower seeds (Ahmad *et al.*, 2010). In the United States about 90% of the biofuel is produced the oil separated from soybeans (Balat & Balat, 2010). Commonly used feedstocks for the synthesis of biodiesel comprises about 59% of rapeseed oil, about 25% of soybean oil, about 10% of palm oil, 5% of sunflower oil and around 1% of some other plant extracted oils (Pahl, 2008). Because of the excessive utilization of these oils extracted from the edible crops or vegetables this led to some nutritional imbalance. For example in the year 2012 around 13% of oils or fats were extracted from edible plants worldwide (Banković-Ilić *et al.*, 2012). Therefore, the consumption of non-edible fats or oils from non-edible plants are more suitable than the oils and fats extracted from edible oil based plants. From this exploration, the *Sesbania bispinosa* (*Sesbania aculeate*) seed oil can be a favorable substitute fuel for diesel engines. It is because of their agricultural origin and lower exhaust emissions without altering the performance of engines. (Mallesham *et al.*, 2020).

Biodiesel being an alternate fuel can minimize our dependence on import suppliers of oils because it can be generated from local feedstocks of energy. Being locally produced, biodiesel has become the major source of the energy by reducing the dependence on fossil fuels and saving a big portion of the budget.

Sesbania bispinosa

Sesbania bispinosa commonly known as Jantar, Danchi, Dunchi or prickly sesbania, is a small tree. It is commonly found in Asian countries like Pakistan, India and North Africa.

It is an annual shrub that has fibrous pithy stem and yellow flowers. *Sesbania bispinosa* produced up to 12 to 19 t/ha/year fodder or manure (Qamar *et al.*, 2014).

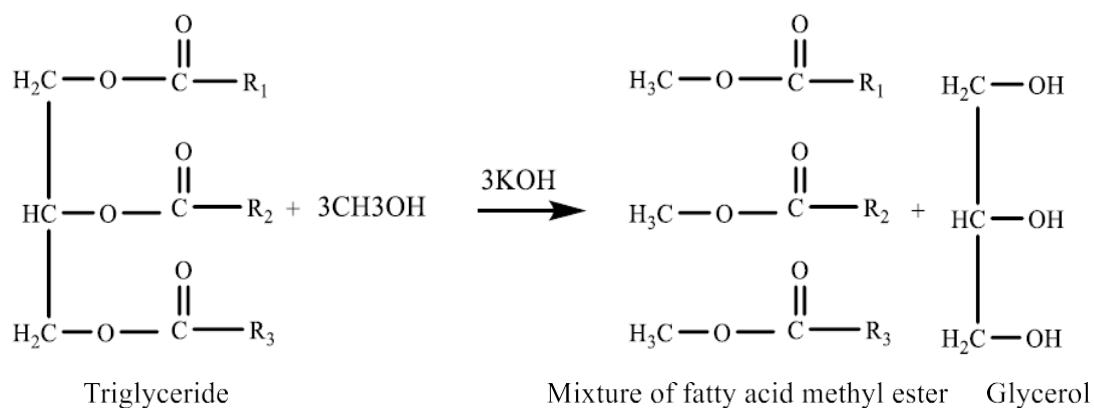
There are about 50 species of sesbania, all having their countless uses in many ways. *Sesbania bispinosa*, also known as *Sesbania aculeate*, is the common specie of the fabaceae family. Lipid extracted by soxhlet method showed higher number of fatty acids in uncooked seeds. (Jeppu *et al.*, 2016)



Sesbania bispinosa seeds (left) plant (right)

Transesterification

Transesterification is the reaction of oil or fat extracted from animal, plant or any other source with an alcohol in the presence of catalyst producing fatty acid alkyl esters (biodiesel) as major product and glycerol as side product (Otera, 1993). This reaction lowers the density of fatty acids by converting them into esters of same fatty acids.



Depending upon the nature of fatty acids present in the oil or fat fuel characteristics can be changed. (Bouaid *et al.*, 2016).

Transesterification require some catalyst that may be of different nature such as alkaline, enzymatic or acid in nature. Most important function of the catalyst is the production of the methoxide ion or to enhance the electrolytic character of the substrate (triglyceride) transferring the equilibrium toward the product formation. (Narasimharao et al., 2007) potassium hydroxide (KOH) or sodium hydroxide (NaOH) are commonly utilized due to easy to handle and less costly. Potassium hydroxide usually results better biodiesel properties as catalyst (Encinar, et al., 2005)

During this process methanol is used commonly due to being inexpensive than ethanol and the easy recovery of unreacted methanol (Zhou and Boocock, 2003).

MATERIAL AND METHODS

Extraction of Oil from *Sesbania bispinosa* Seeds

Seeds of *Sesbania bispinosa* were taken, cleaned and crushed by using electric mixer of high rpm. Oil was then extracted with the help of conventional soxhlet apparatus by using n-Hexane as a solvent. Extracted oil was then filtered and stored.

Transesterification of Oil

2g of KOH (1 %wt of oil) and 55.5 mL of methanol was mixed and added into 200g of oil (heated at about 60 °C) with constant stirring. The reaction mixture was heated for one hour at 60 °C with constant shaking. After one hour of heating the reaction mixture was poured into the separating funnel and left for 12 hour. Then upper layered biodiesel was separated from the lower glycerol layer.

Removal of Soap from Biodiesel

Layer containing biodiesel was washed with distilled water to remove unreacted catalyst, soap, methanol and some leftover glycerol. After the clear separation the lower water layer containing any undesired soap and other impurities was removed from the separating funnel.

This step was repeated (4-5 times) till all the soap was removed from biodiesel and clear upper biodiesel layer was obtained.

Removal of Water Content from the Biodiesel

Sodium sulfate anhydrous was added in the biodiesel and left for some time. Then mixture was filtered to get moisture free biofuel. Removal of water from biodiesel is very necessary because its presence, even in small amount, affect the quality of biodiesel.

Purification of Final Product

In order to remove any water content and methanol the final product was heated at 110 °C for half an hour. After heating, it was cooled and actual yield of pure biodiesel was calculated. After all the purification steps two products were obtained in transesterification e.g. biodiesel (final product) and glycerol (side product). Glycerin formed can be further used for various purposes such as soaps formation, in cosmetics and candle making. Biodiesel formed in this reaction is our desired product that can be utilized as an alternative fuel in comparison to diesel fuel.

Study of Different Factors Affecting the Yield of Biodiesel

This alkaline catalyzed transesterification was studied at different oil to methanol molar ratios (1:4, 1:6, 1:8 and 1:10) in order to optimize the conditions. Similarly different concentrations of potassium hydroxide (0.5%-3.0%) were used to study the effect of catalyst concentration on the transesterification reaction. Different time durations of reaction (15min-120min) were also studied to get maximum yield at constant temperature

Characterization of biodiesel

Biodiesel was characterized with the help of various evaluation tests for the determination of, density, kinematic viscosity, flash point ash content, conradson carbon residue, calorific value, distillation range, sulphur content and acid value with ASTM D6751 test methods and EN 14214.

RESULTS AND DISCUSSIONS

20% oil was extracted from the seeds of *Sesbania bispinosa* and 86% yield of biodiesel was obtained during the transesterification reaction. Free fatty acids were not more than 0.5%.

Effect of methanol concentration on biodiesel yield

It was found that the yield of biodiesel increases with increase in methanol molar ratio up to the ratio of 6:1 (methanol: oil). Any further increase in alcohol ratio resulted in the decrease in fatty acid methyl ester yield due to the high polarity of reaction media, affecting the separation of glycerol from the biodiesel layer. (Figure 1).

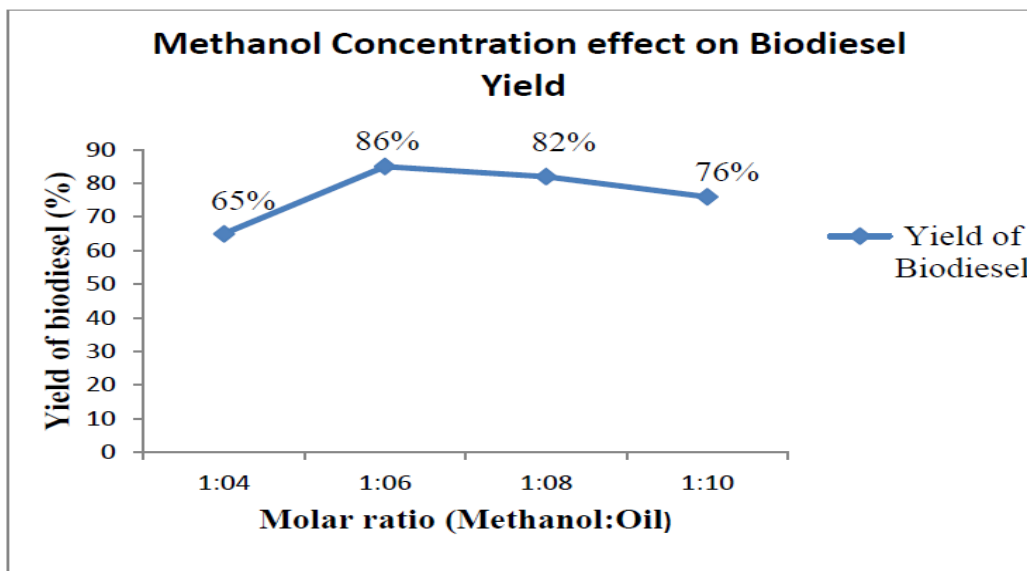


Figure 1: Methanol Concentration effect on Biodiesel Yield.

Catalyst Concentration Effect on Biodiesel Yield

By increasing the amount of potassium hydroxide an increase in the yield was observed with maximum yield of 86% when catalyst amount was 1.0 wt%. Afterwards the yield started to decrease possibly due to the production of greater amount of the soap formation, a major side reaction of the transesterification. A very less catalytic amount was not found to be suitable because it was insufficient for the production of the required amount of methoxide ion, produced when a calculated amount of the methanol and potassium hydroxide was mixed with each other with proper stirring. (Figure 2).

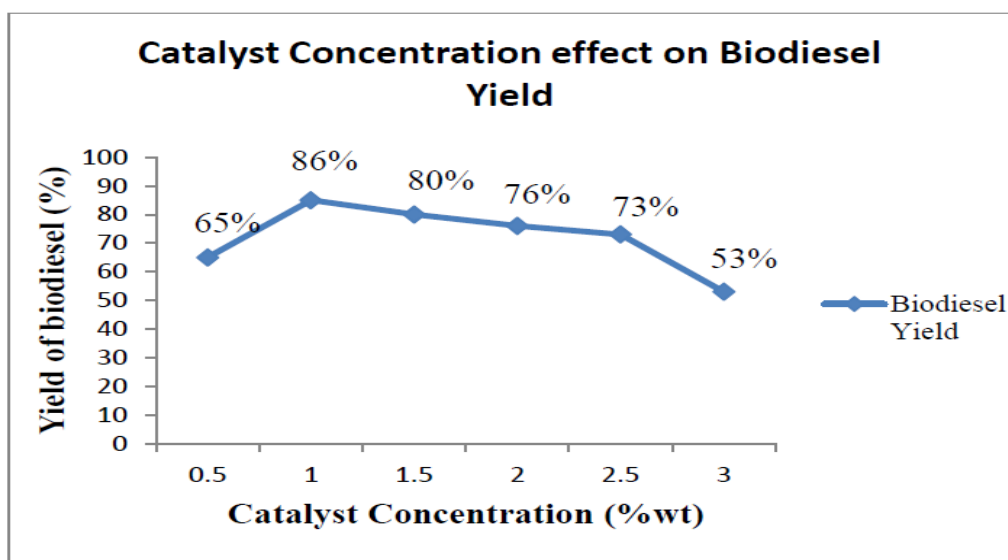


Figure 2: Catalyst concentration effect on biodiesel yield.

3.3. Effect of Time Duration on Biodiesel Yield

As the time period increases the yield of the reaction also increases up to 60 minutes that is optimum duration for the production of biodiesel from *Sesbania bispinosa* seeds oil. After this time period the yield started to decrease possibly due to the reversibility of reaction resulting in the reduction in the amount of biodiesel yield. (Figure 3).

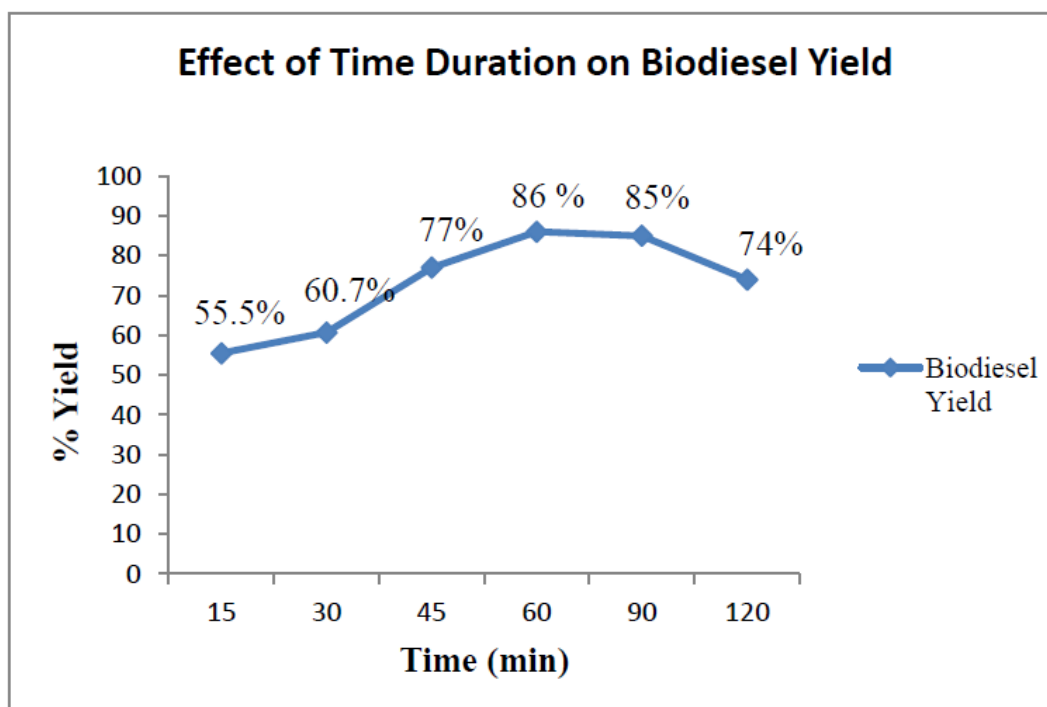


Figure 3: Effect of time duration on biodiesel yield.

Fuel Characteristics

Different fuel characteristics such as density, kinematic viscosity at 40°C, flash point, Conradson carbon residue, ash content, distillation range, calorific value, sulphur content and acid value of prepared biodiesel was examined.

Density and kinematic viscosity

The density of biodiesel was found to be 0.870 g/cm³ and kinematic viscosity at 40°C was 4.76 cSt. These values show that the resulting biodiesel has properties comparable to standard biodiesel values. The low values of density and kinematic viscosity of biodiesel as compare to parent oil make its flow properties better.

Flash point and calorific value

The flash point value was observed to be 177 °C with the help of open cup tester. This high

value of flash point as compare to diesel fuel make biodiesel less hazardous liquid fuel at high temperature. The calorific value of the biodiesel was calculated with calorimetric bomb. The value obtained was 17256.60 btu/lb. The calorific value of biodiesel is lower than the heating value of diesel but is within the limits of values set by authorities for standard fuel characteristics.

Conradson carbon residue and ash content

The value of the conradson carbon residue (CCR) test is 0.0170% that gives information about the coke generating ability of fuels in the engines after the complete burning. In the same way the ash test helps to know about the material (inorganic compounds) that can be abrasive and may become a cause of engine wear. There was no ash left during the performance of this test that means the biodiesel produced in the laboratory is safe for the engine.

Total Sulfur Content and Acid Number

The results of total sulfur content test have value 0.014% that is in very less quantity to harm the environment. High concentration of sulfur (greater than 0.05%) either in gaseous or solid form can affect human health badly. It is the main reason of acid rain having high concentration of sulfuric acid and harming the environment in many ways. The biodiesel could reduce the exhaust emission of harmful gases by 70% than diesel fuel. (Pan, 2011) The acid number of biodiesel that was estimated through titration process was found to be about 0.20 mgKOH/g that was less than 0.50 mgKOH/g, standard value of acid number of biodiesel showing that sample was actually a good biodiesel.

Distillation Range

Distillation of biodiesel was carried out with 100 mL of sample and temperature was noted for different percentage of volume recovered. About 90% of biodiesel was recovered at 329 °C temperature. This value of recovered volume was according to the standard values of distillation obtained by using biodiesel at various temperatures (Figure 4).

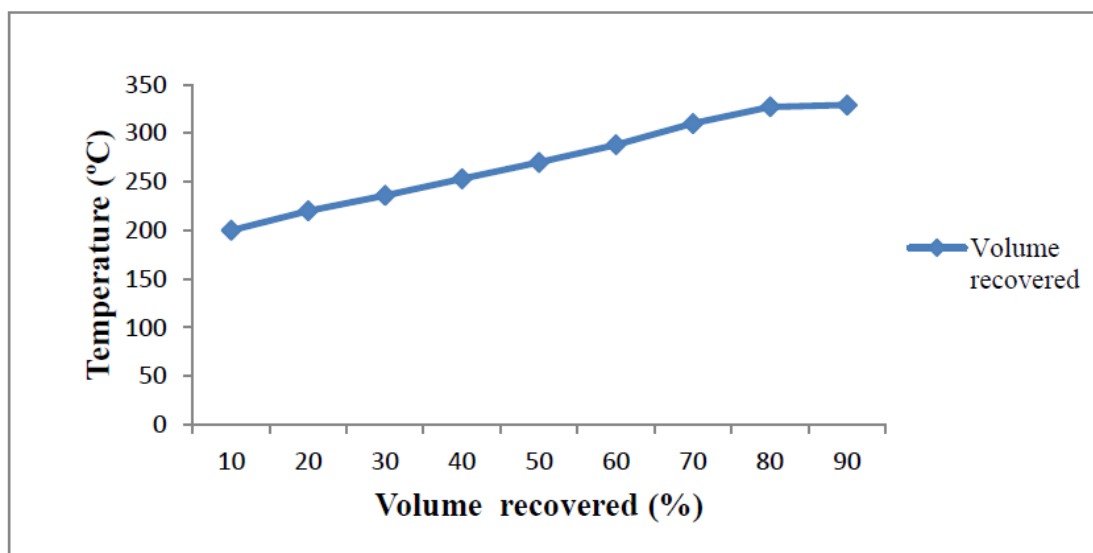


Figure 4: Distillation at different temperatures.

Table 1: Results of all parameters of biodiesel properties.

Properties	Units	Standard values		Biodiesel	Test Methods
		Minimum	Maximum		
Density	g/cm ³	0.869	0.900	0.870	ASTM D1298
Kinematic viscosity at 40 °C	cSt	3.5	5.00	4.76	ASTM D445
Flash point	°C	130	----	177	ASTM D93
Conradson carbon residue	% wt	----	0.05	0.0170	ASTM D189
Ash content	% wt	----	0.020	Nil	ASTM D874
Calorific value	btu/lb	16928	17996	17256.60	ASTM D5865
Distillation range	°C		360	200-329	ASTM D 1160
Sulfur content	% wt	----	0.050	0.014	ASTM D5453
Acid number	mgKOH/g	----	0.50	0.20	ASTM D664

CONCLUSION

Depletion of current limited energy reserves such as fossil fuels is at the peak leading to energy sources deficiency and the severe environmental hazards that should be addressed as soon as possible. In this research biodiesel from the non-edible *Sesbania bispinosa* seeds was prepared with maximum yield of 86%. It was achieved with 1 %wt of KOH, 1:6 oil-methanol molar ratio and reaction duration of 60 minutes heating at 60 °C with constant stirring. The different fuel characteristics were determined and examined to confirm the production of biodiesel such as density (0.870g/cm³), kinematic viscosity (4.76 cSt), flash point (177 °C), conradson carbon residue (0.0170 %wt) ash content (nill), distillation range (200-329°C) calorific value (17256.60 btu/lb), sulfur content (0.014 %wt) and total acid number (0.20 mg KOH/g). All these properties were just in accordance with the limits set by ASTM D6751 standard values of biodiesels. This biodiesel proved to be best substitute of

diesel fuel due to less hazardous to environment and being extracted from renewable sources in nature.

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