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# Optimization of Biodiesel Production Process in *Datura stramonium* Seed Oil, a Non-edible Oil Source

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***Abstract***

Biodiesel is an energy source suggested as an alternative to the diminishing fossil fuels. While research on the screening and identification of suitable energy candidates for biodiesel is on progress, optimization of the production process has to be considered for economic and environmental reasons. Transesterification is the process used for the conversion of triglycerides of oils and fats into fatty acid alkyl esters (biodiesel) and glycerine, in the presence of acid or alkali catalysts. Free fatty acid content, catalyst concentration, alcohol to oil molar ratio, reaction temperature and reaction time are the parameters that influence the production process of biodiesel. The optimization of reaction parameters is the primary step to be assessed in the production process of biodiesel. In the present investigation a detailed study on the optimization of catalyst concentration, alcohol to oil molar ratio, reaction temperature and reaction time has been carried out to determine the maximum conversion of oil to biodiesel, in a non-edible oil source, *Citrullus colocynthis* seed oil. The catalyst NaOH was used in varying proportions of 0.5, 0.75, 1.0 , 1.25 and 1.5 % (w/v ) of oil. The different molar ratios of methanol to oil studied were 3:1, 5:1, 7:1, 9:1 and 11:1. The reaction temperature assessed was between 40° and 90° C. The reaction time was varied from 30 minutes to 150 minutes. The maximum ester conversion in *Datura stramonium* oil was obtained at a catalyst concentration of 1.0% with an alcohol to molar ratio of 7:1 at a reaction temperature range of 60° and 70° C in the reaction time of two hours.

## 1. Introduction

The depletion and scarcity of fossil fuel along with increase in cost of petroleum fuel has advocated interest in finding out alternative forms of energy from renewable resources. Self reliance of energy security of a nation can be attained by utilization of renewable sources of energy, especially the energy from vegetable oils. Being an agricultural country endowed with varied climates, nutrient-rich soil and ability to grow many different crops, India offers a great promise as a producer of surplus raw material for biodiesel. Exploitation of edible oil for biodiesel production is restricted in the country due to its competition for food resources. Non edible oil sources possess a clear opportunity as substitute for fossil fuel in view of economic as well as environmental benefits. According to Azam et al., (2005) the use of edible oil in India is not feasible for the production of biodiesel. Under these conditions only such plants which produce non-edible oils can be considered for biodiesel, and they can be grown in large scale on non cropped marginal lands and wastelands. In India non-edible type oil yielding trees such as linseed, castor, karanja, neem, rubber, jatropha and cashew are in large numbers. The production and utilization of these oils are low at present because of their limited usage (Ramdhas et al., 2005). These sources can serve as substitutes for depleting fossil fuels. Development of biofuels as a renewable energy for transportation is a critical towards achieving self-reliance of energy security.

Biodiesel making process is dependant on variables such as free fatty acid percentage, catalyst concentration, alcohol to oil molar ratio, reaction temperature and reaction time. In the present study the variables affecting the transesterification reaction such as free fatty acid content, catalyst concentration, alcohol to oil molar ratio, reaction temperature and reaction time were systematically investigated in *Datura stramonium* seed oil, a non-edible source to determine the optimal reaction conditions for obtaining higher conversion of oil into biodiesel.

## 2. Material and Methods

The *Datura stramonium* seeds were collected from the areas in and around Coimbatore district, Tamilnadu, India. The extraction of oil was carried out using mechanical expeller. The free fatty acid content in the oil was determined by titremetric method. Two step transesterification process, i.e. acid pretreatment step followed by base catalyzed method was used to produce biodiesel from *Datura stramonium* seed oil.

The effect of alkali concentration on the yield of biodiesel was studied by varying the concentration ranging from 0.5% to 1.5% with 0.25 % increments. The effect of the molar ratio of methanol to oil on the yield of methyl ester formed from methanolysis was studied at various amounts of methanol. The different molar ratios of methanol to oil studied were 3:1, 5:1, 7:1, 9:1 and 11:1. The temperature was fixed at 65° C and catalyst concentration was fixed from the optimization studies. The transesterification reactions were carried out to determine the optimum reaction time (30, 60, 90, 120 and 150 minutes). The biodiesel production was further optimized for reaction temperature by carrying out transesterification process at various temperatures ranges of 40- 50°C, 50-60°C, 60- 70°C, 70-80°C and 80-90°C . The catalyst concentration was fixed at 1.0% and the methanol to oil ratio was fixed at 7:1 ratio for experiments with varying temperature and varying reaction time.

## 3. Results and Discussion

Every oil has specific properties and therefore a unique set of transesterification process parameters. Free fatty acid reduces the yield since the free fatty acids consume the alkali catalyst, because they take part in the neutralization reaction (Turck, 2002; Meher et al., 2006). According to Ma and Hanna (1999), a better conversion could be obtained in a shorter duration with higher alcohol to oil molar ratio. Molar ratio and catalyst concentration and their interaction affect the yield of biodiesel. The reaction stoichiometry involves three moles of alcohol for each mole of triglyceride to obtain three moles of ester and one mol of glycerol. Being an equilibrium reaction, excess alcohol displaces the reaction to ester formation and higher conversions are reached. Nevertheless, a large excess of alcohol makes glycerol separation difficult due to the increasing solubility of glycerol in the alcohol. Moreover, when the glycerol is kept in the solution, it displaces the equilibrium to the left hand side, decreasing the ester yield. Also the yield and optimum rate depend on the oil used.

### Free fatty acid

The free fatty acid is one of the key parameters affecting the yield of biodiesel in transesterification process. The free fatty acid % in the *Datura stramonium* oil sample investigated by titration method was estimated to be 14.1 corresponding to an acid value of 28.2 mg KOH/gm.

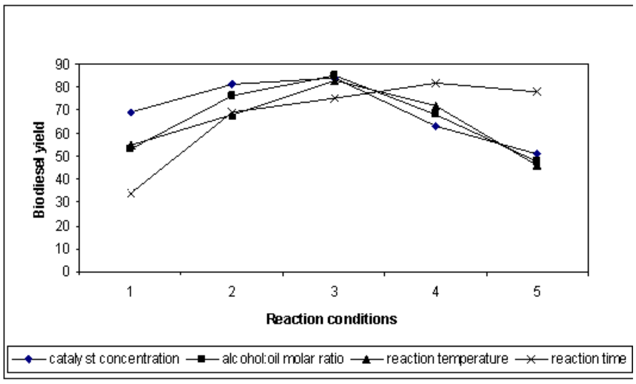
The biodiesel production process depends on the amount of free fatty acid in the oil to be processed. Successful alkaline transesterification requires a free fatty acid value lower than 3 %. One step base catalysed transesterification is applied to feedstock containing a free fatty acid level upto 3%. Freedman and Pryde (1982), Liu (1994), and Mittelbach et al. (1992) have mentioned that the oil should not contain more than 1% FFA for alkaline–catalyzed transesterification reactions. This corresponds to an acid value of 2 mg KOH/g. If the FFA level exceeds this amount, the formation of soap will inhibit the separation of the ester from the glycerin and also reduce the ester conversion rate. In the present study *Datura stramonium* oil was subjected to two step process, a combination of acid esterification followed by base transesterification as FFA level above 3%.

### Influence of catalyst concentration

The effect of catalyst concentration on the product yield was investigated using NaOH as catalyst at different concentrations in the present study. The catalyst amount varied at 0.5, 0.75, 1.0, 1.25 and 1.5 % (w/v) of oil in *Datura stramonium.* The influence of catalyst concentration on the FAME yield in *Datura stramonium* is shown in table 1and fig1. The best results were obtained at a concentration of 1.0 %. For higher catalyst concentration the yield was lower. It has been reported that alkaline catalyst concentration greater than 1.5 % (% wt of oil) leads to the production of large amount of soap (Vincente et al., 1998; Tomasevic and Marinkovic, 2003). The soap can prevent separation of the biodiesel from the glycerine fraction (Demirbas, 2003).

Refined and crude oils with 1% either sodium hydroxide or potassium hydroxide catalyst resulted in successful conversion. Methanolysis of soybean oil with 1% potassium hydroxide gave the best yields (Freedman *et al.*, 1986; Gryglewicz *et al.*, 1999).

Fig 1 : Effect of reaction conditions on the biodiesel yield percentage of *Datura stramonium* seed oil




| Reaction Parameters | 1 | 2 | 3 | 4 | 5 |
|---|---|---|---|---|---|
| Catalyst conc. (w %) | 0.5 | 0.75 | 1.0 | 1.25 | 1.5 |
| MeOH:oil molar ratio | 3:1 | 5:1 | 7:1 | 9:1 | 11:1 |
| Temperature (°C) | 40-50 | 50-60 | 60-70 | 70-80 | 80-90 |
| Time (mins) | 30 | 60 | 90 | 120 | 150 |

Table 1: Effect of reaction conditions on the biodiesel yield percentage of *Datura stramonium* seed oil

| Sl.No. | Catalyst Conc. (w%) | MeOH:Oil Molar ratio | Temperature (°C) | Time (min) | Biodiesel Yield % |
|---|---|---|---|---|---|
| 1 | 0.5 | 6:1 | 65 | 120 | 69 |
| 2 | 0.75 | | | | 81 |
| 3 | 1.0 | | | | 84 |
| 4 | 1.25 | | | | 63 |
| 5 | 1.5 | | | | 51 |
| 6 | 1.0 | 3:1 | 60 | 120 | 53 |
| 7 | | 5:1 | | | 76.2 |
| 8 | | 7:1 | | | 85 |
| 9 | | 9:1 | | | 68 |
| 10 | | 11:1 | | | 48 |
| 11 | 1.0 | 7:1 | 40-50 | 120 | 55 |
| 12 | | | 50-60 | | 68 |
| 13 | | | 60-70 | | 83 |
| 14 | | | 70-80 | | 72 |
| 15 | | | 80-90 | | 46 |
| 16 | 1.0 | 7:1 | 65 | 30 | 34 |
| 17 | | | | 60 | 69 |
| 18 | | | | 90 | 75 |
| 19 | | | | 120 | 82 |
| 20 | | | | 150 | 78 |

**Influence of alcohol to oil molar ratio**

The alcohol to oil molar ratio is one of the most important variables affecting the yield of biodiesel. The stoichiometric molar ratio of methanol to oil for complete transesterification of the fatty acids in the oil to methyl esters is 3:1. However, in practice a higher molar ratio is employed in order to shift the reaction equilibrium toward the products side and produce more methyl esters. In general, the molar ratio is associated with the type of catalyst used.

In the case of *Datura stramonium* the maximum ester yield of 85 % was obtained at a molar ratio of

7:1. Increasing the methanol to oil ratio beyond 7:1 decreased the yield of FAME. (Table 1, Fig. 1).

Freedman *et al.*, (1984) studied the effect of molar ratio (from 1:1 to 6:1, methanol to oil) on ester conversion with vegetable oils such as soybean, sunflower, peanut and cotton seed oils. It was observed that the oils behaved similarly and achieved highest conversions (93-98%) at a 6:1 molar ratio. Tanaka *et al.* (1981) in a two-step transesterification processing of feedstock such as coconut oil, palm oil and tallow used 6:1-30:1 molar ratios with alkali catalyst to achieve a conversion of 99.5%. A molar ratio of 6:1 was used for beef tallow transesterification with methanol, where 80% (by tallow weight) esters was recovered ( Ali 1995). During biodiesel production from vegetable oil, Kim *et al.*, (2004) found that there was no significant increase in biodiesel yield when the alcohol to oil ratios were increased beyond 9:1. A higher molar ratio level resulted in increase in the glycerine phase.

**Influence of reaction temperature**

The effect of different temperatures on the transesterification of *Datura stramonium* is shown in table 1 and fig 1. As expected, higher yield of methyl ester was achieved at a temperature ranging from 60° to 65 °C. It was observed that the yield of biodiesel in all the samples increased steadily upto 60 to 70° C then a gentle decline from 70° to 75° C and steady decrease beyond 75°C. The optimum temperature for maximum biodiesel yield was observed around 65°C. As indicated in the fig.1, at 50°C and 70°C, relatively lower percent yield of methyl esters was evident. With further increase in temperature there was no increase in biodiesel yield. The rate of transesterification rises with increasing temperature (Freedman et al., 1984). However the maximum reaction temperature cannot exceed the boiling temperature of the alcohol used for the study. As the boiling point of methanol is 65° C, the optimal reaction temperature was in the range between 60- 65°C.

Temperature clearly influenced the reaction rate and yield of esters (Ma and Hanna, 1999). The maximum yield of esters occurs at temperatures ranging from 60 to 80°C at a molar ratio (alcohol to oil) of 6:1. Further increase in temperature is reported to have a negative effect on the conversion rate. (Srivastava and Prasad, 2000)

**Influence of reaction time**

The conversion of oil to biodieel approaches to equilibrium conversions with increased reaction times (Meher *et al.*, 2006). In the present study the maximum ester conversion (82%) was obtained at 2 hour reaction time. Similar reaction time has been reported in Madhuca indica (Ghadge and Raheman, 2005), Cypercus esculentus (Ugheoke *et al.*, 2007) and Karanja oil (Srivastava and Verma, 2008). But Ma *et al.*, (1999) have reported that the reaction was very slow during the first minute due to mixing and dispersion of methanol into beef tallow. From 1 to 5 min, the reaction proceeded very fast. The production of beef tallow methyl esters reached the maximum value at about 15 min.

## 4. Conclusion

The biodiesel production from a non-edible oil source , *Datura stramonium* has been investigated and the optimization of the production process has been carried out to determine the required catalyst concentration , methanol to oil molar ratio, reaction temperature and reaction time for maximum biodiesel yield. Two step base catalysed transesterification process was followed for the production process. 82-85 % of *Datura stramonium* oil was converted to biodiesel using 1.0% NaOH catalyst concentration at a molar ratio of 7:1 at a reaction temperature of 65°C and with a reaction time of 2 hours.

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