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Production of Bio Diesel from Soapstock via a Two Step Heterogeneous Catalysis.

Satya Sree N*, Madhusudhan Rao V, and Vijetha P.

VFSTR University, Guntur 522213, Andhra Pradesh, India.

ABSTRACT

The depleting, non-renewable energy sources and enhancing environmental pollution has fuelled the necessity of an alternative energy source. Bio diesel is one such renewable, alternative fuel that has drawn attention due to feasibility in its production from "soap stock", which is a refinery byproduct of vegetable oils and is having high free fatty acid (FFA) content. The production of biodiesel from soap stock (ss) is a viable process since it is not involved in the utilization of edible vegetable oils. It is an ecofriendly fuel due to liberation of low amounts of $CO_2$when compared with petro diesel and hence reduces global warming and its consequences. It is necessary to look about a suitable catalyst that can convert FFA of soap stock into mono alkyl esters on reaction with a monohydric alcohol. So, a two-step heterogeneous catalysis process is selected for conversion of FFA of soap stock into biodiesel via transesterificaction, when treated with methanol in presence of ferric sulphate, which is the catalyst used.

Keywords: Soap stock, Acid oil, Transesterification, FFA, Heterogeneous Catalysis

Corresponding author

INTRODUCTION

Due to increase in the price of petroleum products and the environmental concerns about air pollution, the use of renewable sources as alternative feed stocks for the production of ecofriendly fuel [7] is of growing interest. Bio diesel can serve as an alternative diesel fuel which is derived from the vegetable oils, fats of plants and animals. Bio diesel so far has been mainly produced from edible oils which include soya bean, palm and others. Since the price of edible oils are quite high and usage of these oils as feed stock for bio diesel may affect the food security. Hence it is not viable to produce it from them. Many efforts have been devoted to find cheaper feed stock such as non-edible vegetable oil like Jatropa or waste cooking oils for the production of biodiesel [3].

The economy of any country incredibly depends on energy supplies and hence more dependent for this on fossil fuels. These are depleting day by day and on the other side, environmental problems like high CO_2 emissions as well other particulate emissions goes on increasing. So, to meet the energy requirements, we may have to switch on towards renewable energy sources. To minimize environmental hazards on behalf of usage of fossil fuels, an ecofriendly fuel source is required. To fulfil these two aspects, bio diesel is a suitable solution. As we are using soap stock as the feed stock for the production of bio diesel, we can have a better waste management procedure as soap stock is the refinery waste of crude vegetable oils.

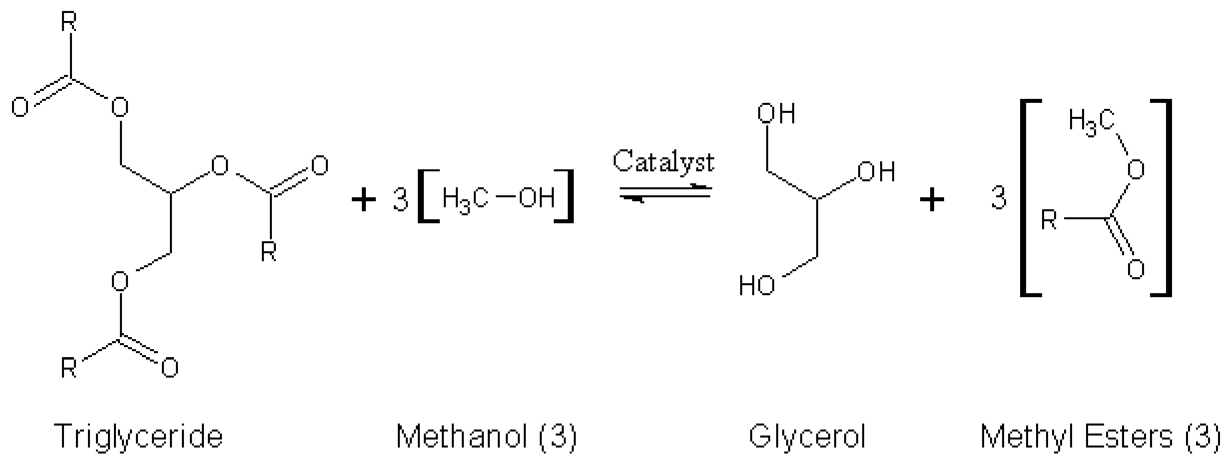
On choosing soap stock, a refinery byproduct of vegetable oils as the potential source of bio diesel production, the task of the work is to convert it into bio diesel by using a catalyst. From this work, we noticed that Ferric Sulphate, being a solid catalyst better serves and the yield of bio diesel is good. The compatibility of solid catalyst for separation of bio diesel is best. So, bio diesel can be obtained from soap stock by a two-step heterogeneous catalytic transesterification process [1-8].

Bio diesel processing from soap stock via two step heterogeneous transesterification gives good yield as the catalyst can satisfactorily convert the FFA of feed stocks without formation of soap.The quality of bio diesel produced should meet the desirable parameters and it is almost on par with the standards of ASTM.

MATERIALS AND METHODS

Soap stock, the potential feed stock for bio diesel production is a heavy alkaline, aqueous emulsion consisting of 10 to 60% of water, 0.1 to 2% sterols & 35 to 85% FFA. Soap stock is generated at the rate of 5% of the volume of the crude vegetable oil refined which equates to an Indian production of approximately 1 million metric tons.

Transesterification Reaction



Skeletal formula of Transesterification

Ferric Sulphate catalysed Methanolysis

It was reported that oils containing high% of FFA cannot be effectively converted into bio diesel by using only alkaline catalyst. So, conversion of soap stock into bio diesel using Ferric Sulphate catalyst via a two-step process is carried out.

- Step I: The mixture of soap stock, methanol & Ferric Sulphate is heated at 90^0C. It is allowed to cool and then centrifuged to separate the catalyst.
- Step II: KOH is added to catalyse the transesterification reaction. The reaction is carried out at 65^0C, and then excess of methanol is recovered under vacuum at 50^0C.

The mixture is left to settle to separate into two layers. The upper oily layer is the bio diesel and unreacted triglycerides. The lower layer is water with insoluble catalyst which can be recovered for further use.

The time schedules followed are 1,2,3 hrs and the weight% of catalyst taken is 1,2,3.

RESULTS AND DISCUSSION

% Conversion

The percentage conversion of soap stock into bio diesel is calculated from the acid value of the layer. The following equation is used.

$$\text{Percentage Conversion} = \left(1 - {AV_{oil}}/{AV_{ss}}\right)\ x\ 100$$

Where AV_{oil} = Acid Value of oil
AV_{SS} = Acid Value of Soap Stock.

With the help of Gas Chromatography equipped with Flame Ionized Detector dried crude bio – diesel samples are analyzed. Nitrogen gas is used as carrier gas. The lauric acid methyl ester is used as reference material.The analysis is performed by dissolving 0.5g of bio – diesel sample and 0.05gm of lauric acid methyl ester into 10ml of n-hexane and injecting 1ml of this solution into the Gas Chromatography.

The purity of the crude Bio – diesel is calculated from the area of sample over the reference area.

The following equation is used.

$$\%\ \text{Purity} = \frac{(Area\ of\ Bio{-}Diesel\ /\ area\ of\ reference\ x\ \ weight\ of\ \ reference)}{(\ weight\ of\ the\ Bio{-}Diesel\ obtained)}\ \text{x}\ 100$$

The samples are withdrawn from reacting mixture at different time intervals and analyzed to know the effect of time on percentage conversion.

Effect of Time

The effect of reaction time on the percentage conversion is shown in the table-1 & fig 1 from the figure it is clear that within an hour the 50% of the free fatty acids were found to be converted in bio-diesel. It also found that almost 85% conversion after 3 hours and no remarkable conversions were noticed even up to 6 hours of time.

Effect of Weight Percentage of Catalyst

The effect of weight percentage of Ferric Sulfate catalyst on percentage conversion is shown in the table-2 & fig 2. The conversion was found to be very slow when no catalyst is used, however upon the addition of 1 wt% of ferric sulfate the percentage raised to 60%. The further increase in amounts 2 wt% and 3 wt% the conversions were found to increase to 90 and 95 respectively.

Effect of mole ratio of methanol to Total Glycerides (TG)

The effect of methanol to TG in mole ratio is given in table –3 and fig-3. When methanol to TG ratio is less than 3, there is a significant effect on conversion. However when the ratio is more than 3 no appreciable effect is noticed.

Table 1: Effect of time on %conversion of soap stock into bio diesel

Sl.No	Reaction Time (hrs)	%Conversion
1	1	50
2	2	80
3	3	85
4	4	85
5	5	85
6	6	85

Table 2: Wt % of catalyst and % conversion

Sl.No	Wt % of Catalyst	% Conversion
1	1	50
2	2	85
3	3	90
4	4	90
5	5	95
6	6	95

Table3: Effect of methanol to TG molar ratio on % conversion

Sl.No	Methanol to TG ratio	%conversion
1	1	56
2	2	74
3	3	92
4	4	96
5	5	96

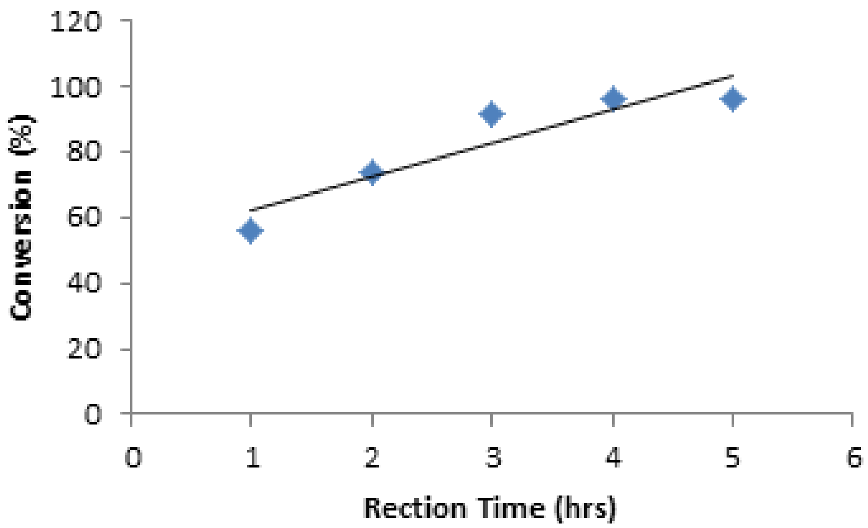


Figure 1: % conversion Vs reaction time

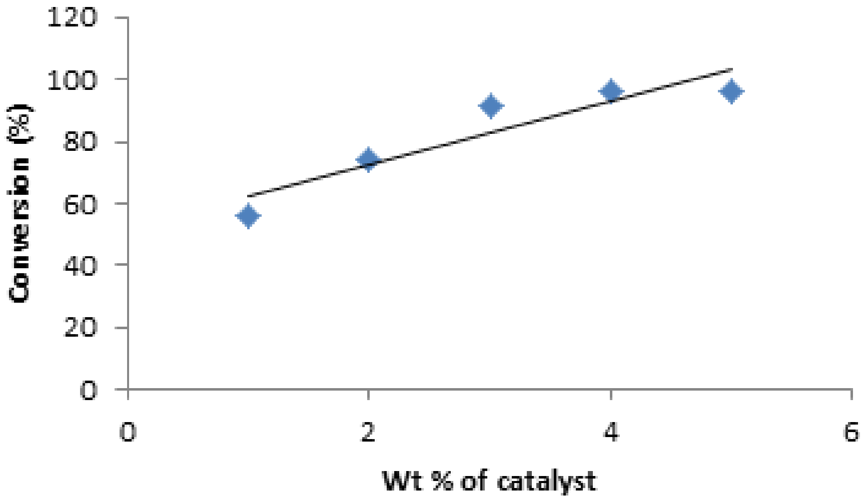


Figure 2: Effect of catalyst weight % on Conversion

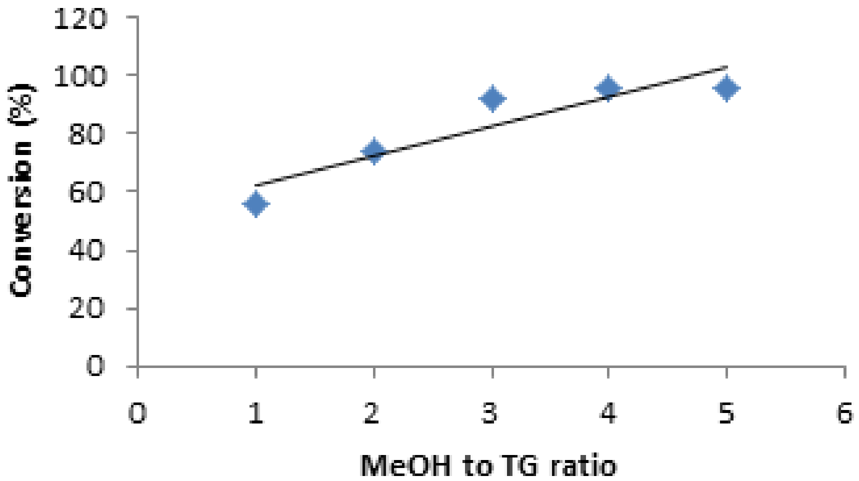


Figure 3: MeOH to TG molar ratio Vs % Conversion

CONCLUSION

The process is a heterogeneous two-step catalysis, where ferric sulfate catalyzed methanolysis. The catalyst is found to be very effective and easy to handle. It can be reused. The optimum conditions are 2 hours' time and 3 wt% of catalyst. If the process is further explored, most of the available Soap Stock can be effectively converted into very useful product i.e., Bio-Diesel, at the same time waste minimization can be carried out in an useful way. The calculated yield of bio-diesel in our studies is not more than 6-8 wt% of Soap stock used. If process is completely optimized, then the yield may go up to a significant level. Hence the process of optimization includes the following aspects:

- Optimization of process conditions: Process conditions like temperature, pressure should be optimized, so that we can increase the yield of bio-diesel.
- Optimization of production cost: Production cost should be minimized , that includes operating cost, accessories cost. And also further studies are necessary to increase the quality of bio-diesel and to scale up the whole process.

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