ID: 2016-ISFT-181

### Production of Biodiesel by Jatropha Oil using Co-Solvent Method

Manjula Jain<sup>1</sup>, Moina Athar<sup>2</sup>

<sup>1,2</sup>Department of Petroleum Studies, Z.H. College of Engineering and Technology, Aligarh Muslim University, Aligarh <sup>1</sup>moinaathar@rediffmail.com

Abstract: The purpose of this research is to provide the information and knowledge about the excellent co-solvent process to produce the high purity biodiesel with minimum reaction time. Transesterification reaction of Jatropha oil with methanol in the presence of catalyst occurs in a heterogeneous system because the reactants are not readily miscible to each other. As a result this leads a longer reaction time and so higher fixed capital investment and product cost due to Transesterification process is batch process. To speed up the reaction rate and to increase the fatty acid methyl ester yield, a mixing solvent Tetra hydro furan (THF) were added, which enables the reaction to bein one phase. The influence of various parameters on the Transesterification reaction including the amount of THF, the amount of catalyst, methanol vol. % and reaction time were investigated. Different tests according to ASTM methods were conducted to check the various properties of produced biodiesel and found according to ASTM D-6751.

*Keywords:* JatrophaCurcas, Transesterification, Tetrahydrofuran (Cosolvent), Biodiesel, Reaction time.

#### **1. INTRODUCTION**

Fatty acid methyl ester (FAME), also known as Biodiesel, derived from renewable feedstock's which falls in the carbon range C12-C22 and have similar properties as diesel. There are various feedstock's which have been proposed for the production of Biodiesel. One of the contending candidates is the JatrophaCurcas, which is a cheaper source of oil for Biodiesel production [1]. Several studies had been conducted on conventional method from the various plant oil through Transesterification with methanol or ethanol and metal hydro oxides as catalyst. Although this conventional method had been found effective in producing the desired quality & yield of Biodiesel but the rate of reaction is very slow often is extending beyond 1 Hr[2]. The main problem for the inferior rate of Transesterificationreaction is that the reaction mixture is not homogeneous because the oils and alcohols are not completely miscible with each other because of their chemical structures [3].Oil disperses in the methanol medium, so the probability and the rate of collision of the glyceride and the methoxide(mixture of methanol and alkaline catalyst (KOH or NaOH) molecules becomes less. This decreases the rate of collisions of molecules and so the rate of reaction causes longer reaction times, increased operating expenses and labor cost [4].

To overcome this difficulty of mixing of the reactants, a single phase reaction is proposed [5]. The proposed model involves a Co solvent introduced in the reaction mixture which makes both the oil and alcohol miscible and reduced the mass transfer resistance. There are various solvents that can be used with the boiling point up to  $100^{\circ}$ C [3-5]. Tetrahydrofuran is preferred because of its boiling point (65°C) similar to that of methanol (64.4°C) so that after reaction, both methanol and THF is recycled in a single step to be used again. The addition of THF to create a single phase greatly accelerates the reaction so that even 97 wt%, is achieved in very short reaction time, such as 10 minutes.

Many researchers have investigated the co-solvent Transesterification of various edible vegetables oils and establishedvarious optimum operating conditions. It has been found that no researchers used JatrophaCurcas oil (non edible) for the production of biodiesel by using THF as co solvent.Therefore in this work Transesterification of Jatropha Oil in The presence of THF as Co Solvent has been carried out.

#### 2. MATERIALS AND METHODS

JatrophaCurcas oil used was brought from "JatrophaVikasSansthan", Delhi. All Chemical reagents used in Transesterification experiments were analytical grade and supplied by Sigma &Merck.

First of all the undesirable impurities like gum and moisture content of Jatropha Oil was removed by filtration and heating the oil at 70°C .After degumming the free fatty acid(FFA) content of oil was determined by titration method. The FFA of oil was found to be more than 5%, exceeding the feasible concentration for base catalyzed Transesterification reaction. To lower the FFA content of the oil acid esterification was performed. Measured quantity of Methanol (60 Vol. %) andSulphuricacid (1Wt. %) were reacted to a known amount of oil. Reaction was carried out at about 55°C under vigorous heating for 1.5 hours. The mixture was then left for separation of layers for 2 hours.

The bottom layer was acid esterified oil that meets the required FFA content (<5%) for base catalyzedTransesterification reaction. initially the reactor was charged with pre -esterified Jatropha oil and heated up to the reaction temperature(55°C). The desired amount of catalyst (KOH) was dissolved in the desired amount of Methanol. In this solution the desired amount of Co-Solvent was added, and the resulting solution was added to the agitated glass reactor containing esterified Jatropha Oil. The reaction was carried out till the desired reaction time. After that, the reaction mixture was placed in the separating funnel and allowed to settle overnight to ensure that the separation of methyl esters and glycerol phases separated completely. Glycerol phase (bottom phase) was removed and kept separately. In order to remove methanol and cosolvent Methyl esters (biodiesel) phase were heated up to their boiling points. Remaining catalyst was extracted by successive washing with hot distilled water. Finally, water present was removed by heating at 110°C.

Optimization of process variables for Transesterification reaction was performed according to the order of following i)best Co-Solvent/ oil Volume %,ii) best reaction time iii)best catalyst (KOH) Concentration and iv)best Alcohol (Methanol) to oil volume %. The best condition for the process variables was determined using maximum Biodiesel yield as criteria. The optimization strategy was hierarchic in principle, i.e. best conditions from earlier optimization levels becomes constant parameters in succeeding levels.KOH was chosen as Catalyst since aside from being cheap, it had been observed to produce the fastest Transesterification reaction at moderate conditions[6].

#### 3. RESULTS & DISCUSSION

#### 3.1 EFFECT OF REACTION TIME ON TRANSESTERIFICATION WITHOUT CO SOLVENT METHOD (FIRST BATCH)





Transesterification reaction of Esterified Jatropha oil has been carried out in the presence of 0.75 wt% KOH catalysts with 20 vol. % methanol at 55°C for varying reaction time from 30-120 minutes. It was found that biodiesel yield increases initially by increasing the reaction time and after 80 minutes it starts decreasing. It was maximum for 80 minutes which was 88%.The reason for decrease of the biodiesel yield after 80 minutes may be because of the backward hydrolysis reaction, which decreases the yield of bio diesel.It was also found that no reaction takes place before 30 minutes because the time was insufficient for the completion of reaction for the given variable combination for the production of biodiesel.

# **3.2 EFFECT OF QUANTITY OF CO-SOLVENT ON TRANSESTERIFICATION WITH CO SOLVENT METHOD (SECOND BATCH):**

Transesterification reaction of Esterified Jatropha oil has been carried out in the presence of 0.75 wt% KOH catalyst with 20 vol. % methanol at  $55^{\circ}$ C for reaction time of 45 minutes by varying THF to oil volume% .It was found that the biodiesel yield increases by increasing the volume % of THF but after 3 vol. % of THF it starts decreasing .the yield is maximum at 3 vol. % of THF.



### Fig. 2. Co-solvent (THF) Volume % V/S Volumetric yield of Biodiesel from Esterified Jatropha Oil using 20 Vol.% Methanol, 0.75 Wt% Catalyst (KOH) at 55<sup>o</sup>C for Reaction time of 45 minutes.

If we compare biodiesel yield with co solvent Transesterification process by without co-solvent Transesterification process, we can see that presence of co solvent even in very small amount (3vol%) increases the bio diesel yield sharply. It is because it makes the system homogeneous that is why biodiesel yield increases as the THF volume % increases. Below 3 vol. %, the reaction mixture did not become fully homogeneous and bio diesel formation was slow but at 3 vol. % the reaction mixture is fully homogeneous and bio diesel yield is maximum. Bio diesel yield slightly decreases after 3 vol. % because of overdilution effect and some biodiesel gets lost due to the presence of extra amount of co solvent during separation process.

#### **3.3 EFFECT OF REACTION TIME ON TRANSESTERIFICATION WITH CO- SOLVENT METHOD (THIRD BATCH)**

Transesterification reaction of Esterified Jatropha oil has been carried out in the presence of 0.75 wt% KOH catalyst, 20 vol. % methanol and 3 vol.% THF(co-solvent)(the quantity that gave the maximum yield in the previous batch) at 55°C for varying reaction time . It was found that as soon as co solvent was added (time=0min) we get 90% yield of bio diesel and if we increase the reaction time further the bio diesel yield also increases up to 5 minutes. This is because solvent makes the reactant oil and methanol miscible and hence homogeneous single phase reaction medium leads to very fast reaction when compared to conventional reaction time. That is why 90% yield is achieved as soon as we add the co solvent after that it increases up to 5 minutes at which the yield is maximum (i.e. 94%) after that it starts decreasing because of backward hydrolysis reaction.



Fig. 3. Reaction Time V/S Volumetric Yield Of Biodiesel Produced From Esterified Jatropha Oil Using 20 Vol. % Methanol, 0.75 Wt% Catalyst (KOH) and 3 Vol. % of THF at 55<sup>0</sup>C.

## **3.4 EFFECT OF CATALYST CONCENTRATION ON TRANSESTERIFICATION WITH CO SOLVENT METHOD (FOURTH BATCH)**

Transesterification reaction of Esterified Jatropha oil has been carried out with 20 vol. % methanol, 3vol. % cosolvent (THF) at 55°C for 5 minutes of reaction time (time at which we got maximum yield of biodiesel in earlier batch) by varying the concentration of catalyst . Biodiesel yield increases initially from 0.25-1 wt%catalyst concentration and we get maximum yield at 1.0 wt% catalyst concentration (97%), after which it starts decreasing. Below 1.0 wt% of catalyst, the quantity of catalyst is insufficient to carry complete out

Transesterification process and above 1.0 wt% catalyst, the amount of catalyst is too high so as to support the saponification reaction and thus biodiesel yield decreases because of soap formation.



Fig. 4.Variation of KOH Concentration V/S Volumetric Yield of Biodiesel Produced from Esterified Jatropha Oil Using 20 Vol. % Methanol, 03 Vol. % THF for 5 Minutes At 55<sup>o</sup>C by Co solvent Method.

#### **3.5 EFFECT OF THE QUANTITY OF METHANOL ON TRANSESTERIFICATION WITH CO -SOLVENT METHOD (FIFTH BATCH):**

Transesterification reaction of Esterified Jatropha oil has been carried out in the presence 1.0 wt. %KOH (catalyst concentration which gave maximum yield in the earlier batch) with 3 volume% co solvent at 55°C for 5 minutes of reaction time by varying the quantity of alcohol (Methanol). Initially by increasing the volume % of methanol,bio diesel yield increases and we got maximum biodiesel yield at 20vol.% which was 97%.Below 20vol.% the alcohol quantity is insufficient to support the forward reaction and hence the bio diesel yield was less. Above 20 vol.% the bio diesel yield decreases, this was because of the excess amount of alcohol was creating problem due to over dilution effect and also during separation some amount of biodiesel get lost during separation process.



Fig. 5.Volume % of Methanol V/S Volumetric Yield of Biodiesel Produced from Esterified Jatropha Oil Using 0.5 Wt% KOH, 3 Volume% THF for 5 Minutes At 55<sup>0</sup>C by Co solvent Method.

#### 4. CONCLUSIONS

In this study, experimental results of a novel method of biodiesel production are introduced and we can conclude that the presence of co solvent makes the reaction fast and increase the yield of biodiesel and also as soon as we added the Co -solvent in the mixture we get 90% yield. Therefore it can be conclusively established that due to the edition of Co-solvent the older batch process becomes continuous.

#### ACKNOWLEDGEMENT

Authors would like to express their sincere thanks to Department of Petroleum studies, Aligarh Muslim University for facilitating this research in its Laboratory.

#### REFERENCES

- [1] Moser, R.; Biodiesel production, properties and feedstock, the society for in vitro biology, springer, pg no: 229-266, (2009).
- [2] Çağlar, Emre.; Biodiesel Production Using Co-

solvent, Department of Chemical Engineering, İzmir Institute of Technology, Urla, Türkiye, European Congress of Chemical Engineering, vol-6, 2007.

- [3] ErwinEscobar, C.; Rex Demafelis, B.; Biodiesel production from JatrophaCurcas oil by transestrification with hexane as cosolvent. Philippine journal of crop science, vol- 33, pg no: 1-13, 2008.
- [4] Guan, G.; Sakurai, N.; Kusakabe, K.; Synthesis of biodiesel from sunflower oil at room temperature in the presence of various co-solvents, Chemical Engineering Journal, 146, 302–6, 2009.
- [5] Encinar, J.M.; González, J.F.; Pardal, A.; Martínez, G.; Transesterification of Rapeseed Oil with Methanol In The Presence of Various Co-Solvents. Third International Symposium on Energy from Biomass and Waste, 2010.
- [6] Vicente, G.; Martinez, M.; Aracil, J.; Integrayted Biodiesel Production: Acomparision of different homogeneous catalyst systems. Bioresourse Technology, 92,297-305, (2004).