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Physico-Chemical Analysis of Biodiesel Derived Glycerin and Saponification of Crude Glycerin with Different Concentrations of Sodium Hydroxide Lye

Doddabasawa[1], Ravikumar Patil[2]

[1]Assistant Professor and Principal Coordinator, Biofuel Information and Demonstration Centre, College of Agriculture, Bheemarayanagudi, Shahapur-Karnataka-585287,

[2]Project Assistant, Biofuel Information and Demonstration centre, College of Agriculture, Bheemarayanagudi, Shahapur-Karnataka-585287,

Abstract: ***Crude glycerin which was obtained during the production of biodiesel with 50 LBP production unit from pongamia seeds was analysed for the free fatty acid, viscosity, pH, color, odour and density and were found that 1.82 %, 118 csts, 8.0, dark brown, unpleasant and 1.5g/cm^3 respectively. The crude glycerin was treated with lye for the preparation of soap with different concentrations. The combination of glycerin (1000ml), water (100ml), sodium hydroxide lye (50g) and raw caustic soda (10g) and lemon juice (50ml) was shown better result with respect to removing of stains, household utensils and oil equipments and pH was in the usability range of 8.0***

Keywords: Crude glycerin, sodium hydroxide lye, biodiesel, transesterification, lemon juice, caustic soda, edible and non-edible seeds.

1. Introduction

Production of Biodiesel is being taken up in the large scale in many parts of the world by using various feed stocks such as vegetable oils, animal fats, edible and non-edible oil seeds, mainly to supplement the fossil fuels. Biodiesel production involves a chemical process in which the tri-glycerides are converted into di-glycerides and mono-glycerides with the help of acid or base catalyst. In this process the esters are separated from the oil. During the process oil is converted into biodiesel (mono methyl ester) and glycerin (C3H8O3).The quantity of glycerin obtained during the biodiesel production varies from 90:10 to 80:20. The ratio depends upon the type and quality of the feed stock.

The glycerin obtained in this process is in crude form and consist of methanol, soap and free fatty acid. This impure glycerin is sold to the lesser prices (***Sims and Bryan, 2011***) and many times left unused, Purified glycerin can be used in the food and pharmaceutical industries but it depends on the availability of purification facility (***J.C. Thompson, B.B. He, 2006***). The purification needs filtration, chemicals, vacuum distillation (***Allen, R.R. et al., 1979***). This process is time consuming and costly process due to this reason most of the time glycerin is unused. Keeping this in lime light an experiment was undertaken to use this raw glycerin for manufacturing of soap with low cost chemicals.

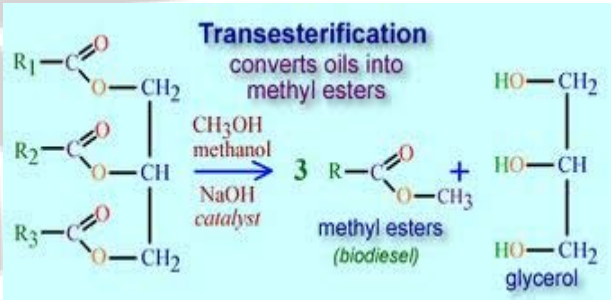

Figure 1: Conversion of oil into biodiesel and glycerin (Source: greener-industry.org.uk)

The composition of the crude glycerin which has been tabulated as in the table:-1.

Table 1: Composition of crude glycerin

Sl. No	*Composition*	*Percentage (%)*
1	Glycerin	50-60
2	Methanol	08-12
3	Methyl esters	15-18
4	Sodium residue as in soap form	12-16
5	Water	02-03

2. Materials and Methods

An experiment was taken up at Biofuel Information and Demonstration centre, College of Agriculture, Bheemarayanagudi-Karnataka-India in the year 2013-14. Pongamia seeds were used as a feed stock for the production of biodiesel, the glycerin which was obtained from the process was used for physico-chemical analysis of free fatty acid by AOAC methods; Ca 5a-40, Viscosity @ 40 ^{0} C MJ800S, density by hydrometer and pH by pH paper ***(Mohammed et., al 2011)***. Later, the soap production on bench scale using the lye sodium hydroxide, raw caustic soda (local market) and the crude glycerin with different concentrations is as detailed below.

- **Concentration 1:** 60gms of sodium hydroxide lye was added in 100 ml of distilled water and it was dissolved completely with glass rod.
- **Concentration 2:** 60gms of sodium hydroxide lye was added in 100 ml of distilled water and it was dissolved completely with glass rod. Later, 50ml of lemon juice was added for fragrance.
- **Concentration 3:** 50gms of sodium hydroxide lye and 10gms of raw caustic soda was added in 100 ml of distilled water and it was dissolved completely with glass rod. Later, 50ml of lemon juice was added for fragrance

The crude glycerin was mixed with the lye solution which contains sodium hydroxide (SDFCL-sd fine chem. Ltd), raw caustic soda (available from the local market-it will make the soaps to hard in short period of time) and lemon juice (fragrance) in different concentrations and was studied for manufacturing of good quality of soaps.

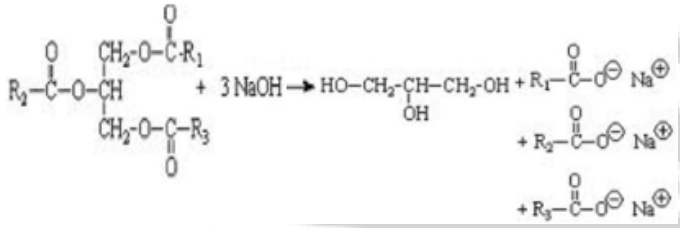

Figure 2: Saponification-conversion of crude glycerin into soap

3. Methodology

1000 ml of crude glycerin which was obtained after the transesterification process of biodiesel was placed in a clean stainless steel tank. The glycerin was heated to 65^0C (because the methanol boiling point is 64.7^0C) to remove the excess methanol present in the crude glycerin, later it was cooled to room temperature. Simultaneously, the lye sodium hydroxide solution was prepared by mixing of 60gms of NaOH in 100ml of distilled water and other ingredients as fragrance and colouring agents and was mentioned in the table no (3). Finally, the lye solution was added to the above cooled methanol removed crude glycerin into the mold with a plastic sheet and allowed to settle for 6 hours. The soap bar was removed from the mold by inverting on a plane good surface. The rectangular shaped soaps were obtained by cutting with knife. The obtained crude glycerin soaps were allowed to cure for 10 days. The pH of the soap was tested by mixing of small piece of soap with distilled water. Later, the pH of the soap was recorded using the pH paper. The same procedure was fallowed for the other two different concentrations of lye-water mentioned in the table no. (3).

4. Results and Discussion

Pongamia pinnata biodiesel derived crude glycerin was analysed for physico-chemical properties as shown in the table (2). It was found that low free fatty acid (1.82%) content in the glycerol. The low content of FFA indicates the purity of glycerol and the rate of conversion of triglyceride into mono glycerides and glycerin. The presence of low free fatty acid in glycerol indicate that the proper conversion of transesterification from P*ongamia pinnata* oil and low content of free fatty acid may maintain the stability of glycerol for longer period. ***Mohammed et., al, 2011*** reported similar low FFA in glycerol obtained from the neem oil.

The viscosity and density of glycerin was 118 csts and 1.50g/cm3 respectively. It is mainly attributed due to presence of methanol and NaOH in the crude glycerol.

The color was found to be dark brown; it may be due to oxidation process. The pH of the crude glycerin was of 9 it was mainly attributed for the presence of methanol and NaOH. It indicates that pH of glycerol was in the formidable range to use the crude glycerol for further value addition.

Results also found that the crude glycerin can be better used by making soap with low cost raw material such as lye and raw caustic soda. It was found that the trail 3rd combination in the table no (3) showed better results with respect to quality, foam and pH value of the soap. The NaOH and raw caustic soda acts as a foaming and binding material. The lemon juice which was added, gave better odour and partial change in the color of the soap.

Table 2: Physico-chemical parameters of the *Pongamia pinnata* biodiesel derived glycerin was mentioned in the table below:

Sl. No	*Parameters*	*Unit*
1	Free fatty acid	1.82 %
2	Viscosity	118 csts
3	pH	8.0
4	Color	Dark brown
5	Odour	unpleasant
6	Density	1.50 g/cm^3

Table 3: Crude glycerin soap with different concentrations

Sl. No	*Description*	*Composition*	*pH*	*Remarks*
1	Concentration I	Glycerin 1000 ml+ 100 ml water+60 gms of NaOH	9.0	Smooth and colour was greenish yellow
2	Concentration II	Glycerin 1000 ml+ 100ml water+60 gms of NaOH+50ml of lemon juice	8.0	Smooth and colour was little brown
3	Concentration III	Glycerin 1000 ml+ 100ml water+50 gms of NaOH+10gms of raw caustic soda+50ml of lemon juice	8.0	Hard and colour was Brown

Figure 3 a): Concentration I

Figure 3 b): Concentration II

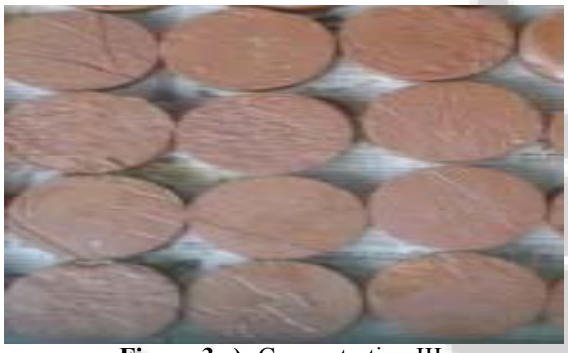

Figure 3 c): Concentration III

5. Conclusion

Crude glycerin which was obtained from biodiesel production process using *Pongamia pinnata* seeds were analysed for physico-chemical properties and found lesser FFA; formidable range of pH, lower viscosity and higher density. The soap was manufactured with the crude glycerin using different concentrations of lye and caustic soda. The results indicated that the crude glycerin can be used as soap. Thus value addition to this unused glycerin which comes as a by-product in biodiesel production may reduce the total cost of biodiesel production.

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Author Profile

Dr. Doddabasawa is working as an Assistant Professor (Forestry) and Principal coordinator (Biofuel Information and Demonstration Centre, Yadagiri District of Karnataka), College of Agriculture, Bheemarayanagudi, Shahapur-Karnataka under the University of Agricultural Sciences, Raichur. He has completed his B.Sc. and M.Sc. forestry in the University of Agricultural Sciences, Dharwad.