

Biodiesel Production from Kapok Seed, Optimization and Characterization to Assess the Suitability of the Product for Varied Environmental Conditions

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Abstract – In this study, non-edible oils (kapok seed) were used to optimize the biodiesel production process variables like catalyst concentration, amount of methanol required for reaction, reaction time and reaction temperature. The fuel properties like specific gravity, moisture content, refractive index, acid value, iodine number, saponification value and peroxide value were estimated. Based on the cetane number and iodine value, The combination of these variables (temperature, catalyst concentration, reaction time, and molar ratio of alcohol to oil) was used in factorial experimental design for biodiesel production optimization by transesterification of Kapok seed oil with methanol.

Index Terms – kapok seed oil, Biodiesel, Catalyst Transesterification, Optimization.

1. INTRODUCTION

Biodiesel is an alternative fuel for diesel engine, where it is produced by chemically reacting a vegetable oil or animal fat with an alcohol such as methanol and ethanol. The most common way to produce biodiesel is transesterification which refers to a chemical reaction between a vegetable oil and an alcohol over a catalyst to yield fatty acid alkyl esters (biodiesel) and glycerol. Triacylglycerol (triglycerides), as the main component of vegetable oil and animal fat, consists of fatty acids with three long chains esterified to a glycerol backbone. When triacylglycerol reacts with an alcohol, three fatty acid chains are released from the glycerol skeleton and combine with the alcohol to yield fatty acid alkyl esters. Glycerol is

produced as a by-product. Methanol and ethanol are the most commonly used alcohol because of their low cost. Both homogeneous and heterogeneous catalysis are used for biodiesel production. The homogeneous catalysts are alkalis such as NaOH or KOH and acid catalysts such as H₂SO₄ or HCl.

The heterogeneous catalyst includes sugar catalyst. The most common that is being developed and used at present is biodiesel, which is fatty acid methyl esters of seed oils and fats and have already been found suitable for use as fuel in diesel engine. Biodiesel is found to be environmentally safe, non-toxic and biodegradable.

Non-edible (pongamia, cotton seed oil and neem oil) oils were used in this experiment. All the oils were first filtered by cloth mainly to remove the dirt and other inert materials from the oil and then placed in a conical flask equipped with magnetic stirrer, thermometer and condenser. Under agitation the raw oil was heated up to nearer to the boiling point to remove the water contaminant present in the oil. After that oil is allowed to cool down under room temperature, and the treated oil alone was taken for biodiesel production purpose. Again, under agitation, the above treated oil was heated up to a desired Temperature on a hot plate. A fixed amount of freshly prepared sodium hydroxide–methanol solution was added into the oils, taking this moment as the starting time of the reaction. When the reaction reached the preset reaction time, heating and stirring

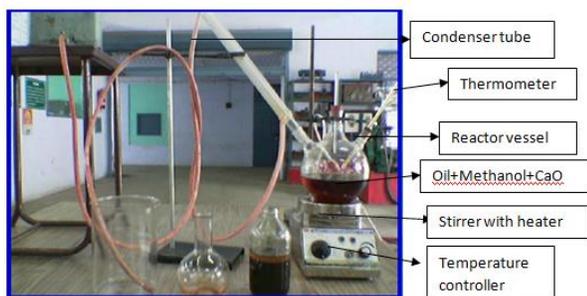
were stopped. The products of reaction were allowed to settle overnight. During settling two distinct liquid phases were formed: crude ester phase at the top and glycerol phase at the bottom.

The crude ester phase separated from the bottom glycerol phase was then washed by cold or warm de-ionized water several times until the washed water became clear. The excess methanol and water in ester phase were then removed by evaporation under atmospheric condition. After that weight of the ester was taken for product yield calculation. The reaction was investigated step by step. The optimal value of each parameter involved in the process was determined while the rest of the parameters were kept constant. After each optimal value was attained, this value was adopted for the optimization of the next parameter. Physical and chemical properties of methyl esters were estimated under laboratory conditions. The cetane number (CN) and higher heating values (HHVs) were calculated from the following equation by using the estimated saponification value (SV) and iodine value (IV).

2. EXPERIMENTAL SETUP AND METHODOLOGY

Transesterification was carried out in a batch reactor, consisting of following components,

- (1). Magnetic stirrer with heater arrangement.
- (2). Single necked round bottomed glass beaker.
- (3). Temperature controller
- (4). Stirrer controller.



Single necked round bottomed glass beaker is used to collect sample of mixture (oil + Methanol + catalyst). Magnetic stirrer and heater provide the stirring and heating effect simultaneously. Temperature controller is used to control the desired heating effect. Stirrer controller is used to control the stirring effect.

Transesterification

Transesterification is a chemical reaction between triglyceride and alcohol in the presence of catalyst. It consists of a sequence of three consecutive reversible reactions where triglycerides

are converted to triglycerides and then diglycerides are converted to monoglycerides followed by the conversion of monoglycerides to glycerol. In each step an ester is produced and thus three ester molecules are produced from one molecule of triglycerides. The transesterification reaction requires a catalyst to split the oil molecules and an alcohol (methanol or ethanol) to combine with the separated esters. Out of these three methods transesterification is the most viable process adopted known so far for the lowering of viscosity. It also gives glycerol as a byproduct which has a commercial value.

Among all these alternatives, transesterification seems to be the best choice as the physical characteristics of fatty acid (m) ethyl esters (biodiesel) are very close to those of diesel fuel and the process is relatively simple. In the esterification of an acid, an alcohol acts as a nucleophilic reagent; in the hydrolysis of an ester, an alcohol is displaced by a nucleophilic reagent. This alcoholysis (cleavage by an alcohol) of an ester is called transesterification. A catalyst is usually used to improve the reaction rate and yield. Because the reaction is reversible, excess alcohol is used to shift the equilibrium to the product side.

Catalyst

A catalyst is needed to improve the transesterification reaction and yield. The alkaline catalysts such as sodium hydroxide, sodium methoxide and potassium hydroxide are most widely used. These catalysts increase the reaction rate several times faster than acid catalysts. Alkaline catalyst concentration in the range of 0.5 to 1% by weight yields 94 to 99% conversion efficiency. Further increase in catalyst concentration does not increase the yield, but it adds to the cost and makes the separation process more complicated.

Reaction Temperature

The rate of the transesterification reaction is strongly influenced by the reaction temperature. Generally, the reaction is carried out close to the boiling point of methanol (60 to 70°C) at atmospheric pressure. With further increase in temperature there is more chance of loss of methanol. Transesterification reaction has been reported to be influenced positively with increase in temperature.

Alcohol to Oil Molar Ratio

The stoichiometric transesterification requires 3 moles of the alcohol per mole of the triglyceride to yield 3 moles of the fatty esters and 1 mol of the glycerol. However, the transesterification reaction is an equilibrium reaction in which a large excess of alcohol is required to drive the reaction close to completion in a forward direction.

The molar ratio of 6:1 or higher generally gives the maximum yield (higher than 98% by weight). Lower molar ratios require a longer time to complete the reaction. Excess molar ratios

increase the conversion rate but leads to difficulties in the separation of the glycerol. At optimum molar ratio only the process gives higher yield and easier separation of the glycerol. The optimum molar ratios depend on the type and quality of the vegetable oil used.

Reaction time

Freedman et al found that the conversion rate of fatty acid esters increases with reaction time. At the beginning, the reaction is slow due to the mixing and dispersion of alcohol into the oil. After a while, the reaction proceeds very fast. Normally, the yield reaches a maximum at a reaction time of <90 min, and then remains relatively constant with a further increase in the reaction time. Moreover, excess reaction time will lead to a reduction in the product yield due to the backward reaction of transesterification, resulting in a loss of esters as well as causing more fatty acids to form soaps.

3. OPTIMIZATION OF PROCESS PARAMETERS

The parameters such as Molar Ratio, Reaction Time and Catalyst Concentration are determining the biodiesel production. Using Response Surface Methodology (RSM) and Taguchi Orthogonal Array process parameters Molar Ratio, Reaction Time and Catalyst Concentration are optimized.

3.3.1 Response Surface Methodology (RSM)

The reaction variables influencing the biodiesel yield that are taken in this work are molar ratio, catalyst concentration and reaction time.

The effect of these parameters on the biodiesel yield were analysed by RSM. Response Surface Methodology is a collection of mathematical methods that are useful for the modelling and analysis of engineering problems. The main objective of this technique is to optimize the response surface that was influenced by various process parameters. Response surface methodology also quantifies the relationship between the controllable input parameters and the obtained response surfaces.

RSM helps in,

1. Understanding the influence of test variables, which normally affect the selected process response
2. The determination of the interrelations among the test variables
3. The characterization of the combined effects that all the test variables which may have influence on the process response.

The Design Expert 6.0 software was used to design the process parameter and the response is statistically analyzed and validated to derive objective inferences.

The experimental design as a function of the selected process variables were carried out using Central Composite Rotable Design (CCRD). Once the variables having the greatest influence on the responses were identified, the Central Composite Design was used to optimize the levels of these variables. It's well suited for fitting a quadratic surface, which usually works well for process optimization.

Water Heating

Suitable amount of water was taken into a container flask and heated till temperature rises to 70°C.

4.2.2 Purification of Kapok seed oil

Suitable amount of Kapok soil was added into container flask containing hot water, and wait for 30 minutes till impure particles settles down.

4.2.3 Separation of Kapok seed oil

Hot water containing Kapok seed oil was collected into the separating funnel and shaken it vigorously for 5 to 10 minutes. Because of low density of Kapok seed oil settles on the top of the funnel and high density water and impurities settled at bottom of the funnel. Then disperse water and impure particles by opening the funnel valve, after that the purified Kapok seed oil was collected into the jar.

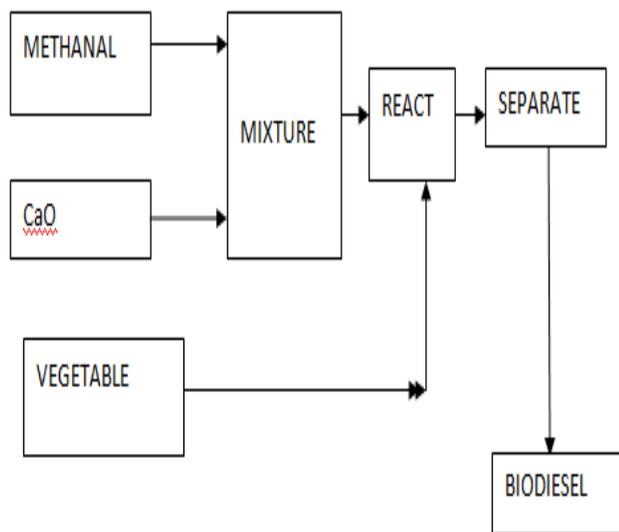


Removal of moisture from purified Kapok seed oil

Collected purified Kapok seed oil was heated to 65°C to remove moisture contained in oil.

4. EXPERIMENTAL PROCEDURE

The below flow chart shows the processing steps for biodiesel production



FLOW CHART FOR BIODIESEL PRODUCTION

Transesterification reaction assays and analysis

The transesterification reaction was carried out with methanol/oil ratio (i.e., 3:1, 6:1, 9:1, 12:1 and 15:1 M), using 0.3 wt%, 0.5wt%, 0.7wt% and 1 wt% of CaO, as a base catalyst. The reactor shown above was preheated, and then the oil was added. When the oil reached the temperature established for the reaction, the methanol and catalyst was added, in amounts established for each experiment.

The reaction was carried out at 50, 55, 60, and 65°C and the reaction times were 2, 2.5, 3, and 3.5h. The choice of the said variables and their range were selected based on several outside sources and on preliminary studies in our laboratory. The combination of these variables (temperature, catalyst concentration, reaction time, and molar ratio of alcohol to oil) was used in factorial experimental design for biodiesel production optimization by transesterification of Kapok seed oil with methanol

Separation Process

The ester/glycerol separation is typically the first step of product recovery in most biodiesel processes. The separation process is based on the facts that fatty acid alcohol esters and

glycerol are sparingly mutually soluble, and that there is a significant difference in density between the ester and glycerol phases. Glycerol is heavier than biodiesel.

The settling will begin immediately, but the mixture is left a minimum of four to five hours. The difference in viscosity and colour between the two liquids can be seen.

The objective is to remove only the Glycerol and stop when the biodiesel is reached. Two phases (having different density) are formed as a result of trans-esterification. Separation is done using a separating funnel.

Upper layer consists of bio-diesel, alcohol, and some soap (formed as a result of side reaction saponification - free fatty acids get converted to soap). Lower layer consists of glycerin, excess alcohol, catalyst, impurities, and traces of unreacted oil. Purification of upper layer (to obtain biodiesel) is done in two steps.

Removal of alcohol – by keeping mixture at elevated temperature ~80 °C. Removal of saponified products – by washing with warm water. Water is immiscible with bio-diesel, hence can be easily separated from bio-diesel.

Washing Process

The primary purpose of the ester washing step is the removal of any soaps formed during the transesterification reaction. In addition, the water provides a medium for addition of acid to neutralize the remaining catalyst and a means to remove the product salts.

The use of warm water prevents precipitation of saturated fatty acid esters and retards the formation of emulsions with the use of a gentle washing action. Gentle washing prevents the formation of emulsions and results a rapid and complete phase separation. The phase separation between esters and water is typically very clean and complete. However, the equilibrium solubility of water in esters is higher than the specified water content.

Therefore, after the washing step there will be more than the equilibrium amount of water present. Water was added with the Biodiesel and it was mixed. Water was drained out and Indicator Phenolphthalein was added. This washing was repeated until the drained water maintains the pink colour.

Drying

The final product of purified ester was heated to 110°C under vacuum to remove residual moisture around three hours to heat by using oven. Then the purified biodiesel is obtained. Many of the parts in the diesel fuel injection system are made of high carbon steels, thus they are prone to corrosion when in contact with water. Hence it is necessary to dry the water washed biodiesel product.

Factorial experimental design and optimization of parameters

Temperature, methanol-to-oil ratio, catalyst concentration and reaction time were chosen as independent variables and the production of methyl esters as dependent variables.

The experimental range and levels of independent variables for bio diesel production are given in table 4.1. A 2^4 full- factorial BBD for four independent variables at five levels was employed and the total number of experiments was 29 ($= 2^k + 2k + 5$).

| Variables | Symbol coded | Units | Range | |
|------------------------|----------------|-------|-------|------|
| | | | Low | high |
| Molar Ratio | X ₁ | Mole | 3 | 15 |
| Catalyst concentration | X ₂ | Wt% | 0.3 | 1 |
| Temperature | X ₃ | °C | 55 | 65 |
| Time | X ₄ | H | 2.5 | 3.5 |

Experimental range and levels of the independent variables

The Design Expert 8.0 software was used for regression and graphical analyses of the data obtained. The maximum values of the yield were taken as the responses of the design experiment. Statistical analysis of the model was performed to evaluate the analysis of variance (ANOVA).

Full factorial Box Behnken design matrix for biodiesel production. Once the experiments are performed, the response variable (conversion to biodiesel) was fitted to a second order model in order to correlate the response variable to the independent variables. The general form of the second- degree polynomial equation is as follows

$$y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i>j}^k \sum_j \beta_{ij} X_i X_j + \epsilon$$

Where i and j are the linear and quadratic coefficients respectively, β is the regression coefficient, k is the number of factors to be studied and optimized in the experiment and ϵ is the random error.

| Run | Independent variables | | | | Response |
|-----|-----------------------|----------------|----------------|----------------|----------|
| | X ₁ | X ₂ | X ₃ | X ₄ | Yield % |
| 1 | 3 | 0.3 | 60 | 3 | 62 |
| 2 | 3 | 0.65 | 60 | 3.5 | 65 |
| 3 | 9 | 0.65 | 65 | 2.5 | 86 |
| 4 | 9 | 0.3 | 55 | 3 | 82 |
| 5 | 3 | 0.65 | 60 | 2.5 | 63 |
| 6 | 9 | 1 | 55 | 3 | 80 |
| 7 | 15 | 1 | 60 | 3 | 76 |
| 8 | 3 | 0.65 | 65 | 3 | 69 |
| 9 | 9 | 0.65 | 60 | 3 | 94 |
| 10 | 15 | 0.65 | 65 | 3 | 78 |
| 11 | 9 | 0.65 | 60 | 3 | 95 |
| 12 | 9 | 0.65 | 60 | 3 | 94 |
| 13 | 15 | 0.65 | 60 | 3.5 | 74 |
| 14 | 3 | 1 | 60 | 3 | 67 |
| 15 | 9 | 0.65 | 60 | 3 | 95 |
| 16 | 9 | 0.65 | 60 | 3 | 94 |
| 17 | 15 | 0.3 | 60 | 3 | 72 |
| 18 | 15 | 0.65 | 60 | 2.5 | 73 |
| 19 | 9 | 0.65 | 65 | 3.5 | 91 |
| 20 | 9 | 1 | 65 | 3 | 88 |
| 21 | 9 | 0.3 | 60 | 3.5 | 84 |

5. RESULTS AND DISCUSSION

5.1. Regression analysis

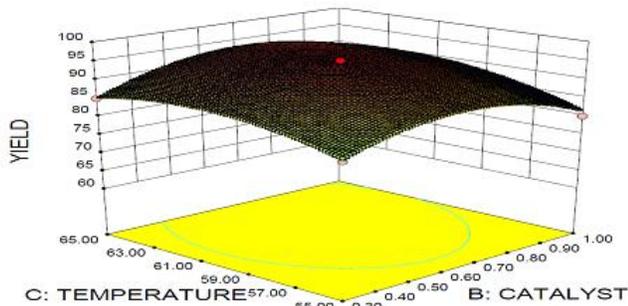
If there is a functional relationship between the two variables X and Y the points in the scatter diagram will cluster around some curve called the curve of regression. If the curve is a straight line it is called line of regression between two variables.

If we fit a straight line by the principle of least squares to the points of the scatter diagram in such a way that the sum of squares of distance is parallel to the Y-axis, from the points to the line is minimized. We obtain a line to best fit for the data and are called regression line of Y on X. Regressions are of two types a) Simple regression b) Multiple regression Simple regression: The relationship between one independent variable and one dependent variable Multiple regression: The

relationship between two or more independent variables and one dependent variable.

The response surface contours, which are the graphical results of interactive effects, are shown in below Figs. Shows that methyl ester conversion increases when high catalyst concentration is applied. However, it was observed that temperatures greater than 62°C and catalyst concentrations greater than 0.7 wt % led to the production of large amount of soap in this study. Furthermore, the addition of an excessive amount of catalyst increases emulsion formation.

When kapokseed oil and CaO dissolved in methanol, there are two reaction pathways: transesterification to produce biodiesel and saponification to produce soap; forecasting the temperature effect is not straightforward. There are two equilibrium reactions. When saponification is favoured, CaO is lost and overall process rate decreases. CaO is a catalyst on transesterification reaction and a reagent on saponification reaction. On the other hand, the transesterification reaction can be favoured when adequate temperatures are used. Low temperatures decreases the saponification rate, thus transesterification reaction is favoured. When temperature increases, the reaction rates are obviously higher because molecules have more energy, but the saponification reaction rate speeds up, therefore the transesterification reaction yield decreases.



Response surface contour interaction on methyl esters production between temperature and catalyst concentration Chemicals and reagents

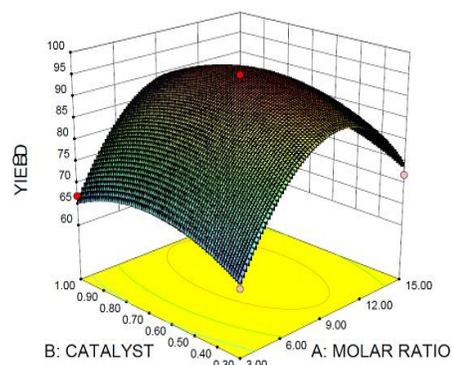
Therefore temperature was tested as an important variable to enhance the reaction in biodiesel production. To summarize, defining the best temperature is clearly an optimization problem.

Shows higher production of methyl esters is strongly favoured when higher molar ratio is employed for a certain time of reaction. The molar ratio is a fundamental variable in the transesterification of the biodiesel production. The said molar ratio affects the separation and recovery of glycerol. A molar ratio of 6:1 is generally considered the most appropriate for ethanol, although in this work, as been indicated, it was found to be molar ratio 9:1 to be best for methanol.

Some researchers have hypothesized that the quality of esters depends on the large excess of alcohol. However, in this study, the high molar ratio of alcohol to vegetable oil interfered in the glycerol separation, since there was increase in solubility. When glycerol remains in solution it helps driving the equilibrium back to the left, thereby lowering yield of esters. The transesterification of cottonseed oil with methanol was observed at molar ratio between 3:1 and 15:1

For molar ratio less than 6:1, the reaction incomplete. For a molar ratio of 15:1 the glycerol separation was difficult and the apparent yield of esters decreased, since some of the glycerol remained in biodiesel phase. Therefore, molar ratio 9:1 seems to be the most appropriate. Several researchers have found similar results for biodiesel production.

Shows the methyl ester yield as a function of molar ratio and catalyst concentration under experimental conditions defined by factorial design. It is possible to observe that higher methyl ester yield occur at high molar ratio greater than 12:1 and at high catalyst concentration greater than 0.7 wt%. When molar ratio values were very low less than 6:1 yields increased with higher catalyst concentration. However, when molar ratio was kept in its higher level (15:1) a higher methyl ester was always achieved. Thus, catalyst concentration is the most important factor in improving methyl ester yield. But an excess catalyst can produce emulsions and the biodiesel that is produced has difficulty in the separation phase.



It has been reported that low catalyst concentration increases conversions with ethanol-oil-ratio. In this present study applying response surface design, we observed that for methanol, methyl esters production increases when catalyst concentration increases for low molar ratio.

The optimum values for selected variables were obtained by solving the regression equation. (2). The optimum values of the process variables for maximum methyl esters production are shown in table 5.3 when a yield of 95% was achieved. The ester yield increased as the molar ratio also increased up to a value 12:1. The best results were for molar ratios between 9:1 and 12:1. The reaction was incomplete for molar ratios less than

6:1. For a molar ratio of 15:1, the glycerol separation was difficult and the apparent yield of esters decreased, because of a portion of the glycerol remaining in the biodiesel phase. Therefore, the phase separation becomes more difficult when molar ratio of methanol increases due to its miscibility increasing for both phases (glycerol and methyl ester).

| Parameters | Optimum values |
|-------------------------------|----------------|
| Ethyl esters (%) | 95 |
| Molar ratio(moles) | 9:1 |
| Catalyst concentration (wt %) | 0.7 |
| Temperature ($^{\circ}$ C) | 60 |
| Time (h) | 3 |

Optimum values of the process parameter for maximum efficiency

The optimum- value temperature was 60 $^{\circ}$ C. This temperature is below the boiling point of alcohol. Therefore, molar ratio is used between 6:1 and 12:1, since it did not evaporate. The transesterification rate increases as the temperature increases. However, the maximum operating temperature should not exceed the boiling point of the reactants.

The highest methyl ester yields were achieved when the reaction time was 3h. Methanolysis is quicker than ethanolysis. The optimum value of catalyst concentration was at 0.7wt%. As a typical catalyst concentration for transesterification reactions (0.3 to 1 wt %), the results that were obtained agreed with those obtained from the response surface analysis, confirming that RSM was effectively used to optimize biodiesel production.

Gas chromatography

The purpose of Gas Chromatography analysis is to know the fatty acid composition present in the oil or biodiesel. The purity of a substance also can be found out. A gas chromatograph is a chemical analysis instrument for separating chemicals in a complex sample.

A gas chromatograph uses a flow-through narrow tube known as the column, through which different chemical constituents of a sample pass in a gas stream (carrier gas, mobile phase) at different rates depending on their various chemical and physical properties and their interaction with a specific column filling, called the stationary phase. As the chemicals exit the end of the column, they are detected and identified electronically. The function of the stationary phase in the column is to separate different components, causing each one to exit the column at a different time (retention time). Other parameters that can be used to alter the order or time of

retention are the carrier gas flow rate, column length and the temperature. In a GC analysis, a known volume of gaseous or liquid analyte is injected into the "entrance" of the column, usually using a micro syringe. As the carrier gas sweeps the analyte molecules through the column, this motion is inhibited by the adsorption of the analyte molecules either onto the column walls or onto packing materials in the column.

The rate at which the molecules progress along the column depends on the strength of adsorption, which in turn depends on the type of molecule and on the stationary phase materials. Since each type of molecule has a different rate of progression, the various components of the analyte mixture are separated as they progress along the column and reach the end of the column at different retention time. A detector is used to monitor the outlet stream from the column; thus, the time at which each component reaches the outlet and the amount of that component can be determined. Generally, substances are identified (qualitatively) by the order in which they emerge (elute) from the column and by the retention time of the analyte in the column. The chromatogram of undistilled and distilled biodiesel. With respect to various retention times, the intensity shown on flame ionization recorder is noted. The following table shows the fatty acid composition of palm stearin, undistilled and distilled biodiesel.

| Composition | Fatty acid (%) | | |
|-------------------------------------|----------------|-----------------------|---------------------|
| | Kapok seed oil | Undistilled Biodiesel | Distilled Biodiesel |
| Lauroic acid (C ₁₂) | 0.86 | 3.16 | 4.74 |
| Myristic acid (C ₁₄) | 1.89 | 2.47 | 4.61 |
| Palmitic acid (C ₁₆) | 58.64 | 49.96 | 61.4 |
| Stearic acid (C ₁₈) | 4.36 | 3.56 | 1.85 |
| Oleic acid (C _{18:1}) | 28.87 | 30.01 | 18.6 |
| Linoleic acid (C _{18:2}) | 3.61 | 7.96 | 0.12 |
| Linolenic acid (C _{18:3}) | - | 5.81 | 4.07 |

Fatty acid compositions

Shows the fatty acid compositions present in cottonseed oil, undistilled and distilled biodiesel. In this result, fatty acid compositions are decreasing from cottonseed oil to biodiesel and it is further decreasing when it is distilled.

From this it can be concluded that the FFA content of the cottonseed oil is decreased when it is converted into methyl ester. When FFA content is less, it helps to have low viscosity. It also helps to have low cloud point and pour point.

6. CONCLUSION

The present study demonstrates that microwaves can be effectively used for biodiesel production from kapok seed oil,

with the major advantages of considerable reduced amount of catalyst and reduced reaction time. The effect of molar ratio, catalyst concentration, reaction time was studied. The reaction parameters were optimized by using design expert software. The more dominant variables that affected transesterification of kapok seed oil to biodiesel were molar ratio, reaction time followed by catalyst concentration. Microwave irradiation significantly decreased the reaction time as compared to conventional transesterification process. The conditions to get optimal response with 88.20% of FAME yield were found to be, 9.50 moles for methanol to oil ratio, 9;1 Wt. % of catalyst and 2.5h for reaction time.

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