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Abstract: The scarcity of the fossil fuel, environmental pollution and food crisis are the world's major issues in current era. Biodiesel is an alternative to diesel fuel, environment friendly and biodegradable and is produced from either edible or non-edible oils. In this study, a non-edible rubber seed oil (RSO) with high free fatty acid (FFA) content of 45% were used for the production of biodiesel. The process comprises of two steps. The first step is the acid esterification to reduce the FFA value and the second step is the base transesterification. The response surface methodology (RSM) was used for parametric optimization of the two stage processes i.e. acid esterification and base transesterification. The yield of biodiesel was analyzed using gas charomatrogphy. The FTIR spectrum was also determined to confirm the conversion of fatty acid to methyl esters. The fuel properties were tested using the standard procedure of ASTM D6751 and EN14214.The properties were within the ranges of the biodiesel standards.

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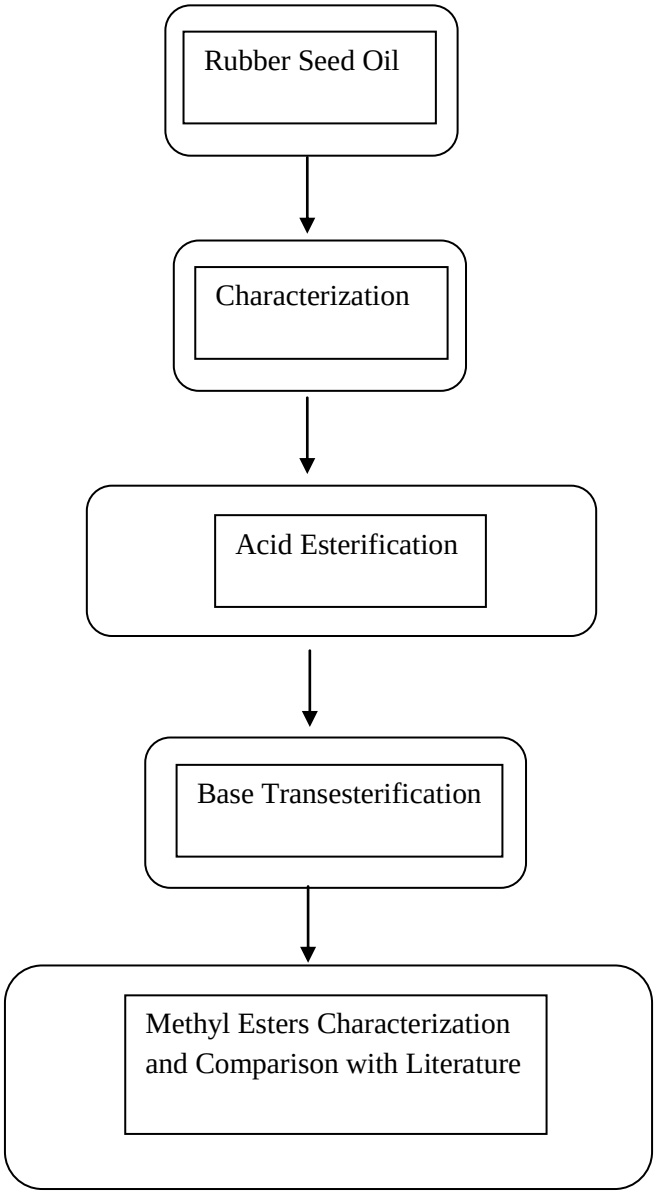

Highlights

- This article presents the comparative studies of the fuel properties of rubber seed oil based biodiesel.
- The design expert has been adopted for the optimization of the process variables.
- The FTIR, cold flow properties and oxidation stability findings of the present study and compared with previous studies and proved to be improved.
- The current work lighten the optimize approach in reducing the FFA values of rubber seed oil based oil biodiesel proved which is economical.
- Conventional method followed the consecutive steps to reduce the FFA values.
- All the fuel properties meet the standards such as ASTM D6751 and EN 14214.

Cover letter

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May 30, 2013

Prof. Moh'd Ahmad Al-Nimr
Editor in Chief
Journal of Energy Conversion and Management

Subject: Submission of an article for publication in Journal of Energy Conversion and Management

Dear Editor,

On behalf of my co-authors and myself, I am submitting the enclosed article entitled "**Comparative Studies of Fuel Properties of Rubber Seed Oil Based Biodiesel**". The manuscript has not been submitted for publication nor has it been published before in whole or in part elsewhere. The authors are in the opinion that the enclosed article fits well within the scope of **Journal Energy Conversion and Management**. This article presents the comparative studies of the fuel properties of rubber seed oil based biodiesel. The design expert has been adopted for the optimization of the process variables. The FTIR, cold flow properties and oxidation stability findings of the present study and compared with previous studies and proved to be improved. The current work lighten the optimize approach in reducing the FFA values of rubber seed oil based oil biodiesel proved which is economical. Conventional method followed the consecutive steps to reduce the FFA values. All the fuel properties meet the standards such as ASTM D6751 and EN 14214.

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I thank you in advance for considering our article for publication in your journal and I look forward to hearing from you.

Sincerely,
Dr Suzana Yusup

Comparative Studies of Fuel Properties of Rubber Seed Oil Based Biodiesel.

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ABSTRACT

The scarcity of the fossil fuel, environmental pollution and food crisis are the world's major issues in current era. Biodiesel is an alternative to diesel fuel, environment friendly and biodegradable and is produced from either edible or non-edible oils. In this study, a non-edible rubber seed oil (RSO) with high free fatty acid (FFA) content of 45% were used for the production of biodiesel. The process comprises of two steps. The first step is the acid esterification to reduce the FFA value and the second step is the base transesterification. The response surface methodology (RSM) was used for parametric optimization of the two stage processes *i.e.* acid esterification and base transesterification. The yield of biodiesel was analyzed using gas charomatrogphy. The FTIR spectrum was also determined to confirm the conversion of fatty acid to methyl esters. The fuel properties were tested using the standard procedure of ASTM D6751 and EN14214.The properties were within the ranges of the biodiesel standards.

Keywords: Biodiesel, Rubber Seed Oil, RSM, Optimum Parameters, Biodiesel fuel quality

1. Introduction

In the last few decades world scarred by dwindling of fossil fuels and its limited resources for future. The world's energy consumption mainly depends on the liquid and solid fossil fuels. Fossil fuel reserves are few in numbers and they are already reaching their peak production. These non- renewable resource consumption rates are faster than the production. To save the world for future energy crises the renewable energy sources are more attractive option [1]. All the renewable energy technologies are eco-friendly and sustainable. Currently renewable energy source adoption faced hurdles because of economic problems, shortage of supply, and lack of technical aspects [2].The fossil fuels and their products are the major contributors of the greenhouse gases, global warming , air pollution, incomplete burning of hydrogen, carbon and particulate matter. Renewable energy sources are the ideal solution of these problems [3]. Biodiesel is defined by the ASTM (American) as a liquid fuel that composed of the fatty acid alkyl ester of the long chain fatty acid derived from the vegetable oil and animal fat. Generally there are four major types of feedstock available for the biodiesel production including oil seed (vegetable oil), animal fats, algae and different low quality material such as waste cooking oil, greases and soap stock[1,4]. Biodiesel production at industrial scale mostly utilized oil such as soybean, palm and canola. But the excess use of the vegetable oil (edible oil) leads, to food versus fuel crisis. The high cost of biodiesel is one of the major hurdle towards its large scale commercialization. About 80% or more of biodiesel cost is altered by its feedstock price [5]. Present researcher are focusing on the non-edible oil sources for the biodiesel production, such as jatropha, moringa olerfera, Pongamia pinnata and camelina sativa [6-9].

Current study utilizes the non edible rubber seed oil for the biodiesel production. Rubber tree (*Hevea brasiliensis*) belongs to the family of euphorbiaceous. The oil content in rubber seed

is between 40 to 50 % [10]. Malaysia is one of the major rubber producing country in the world, according to Association of Natural Rubber Producing Countries with an estimated rubber seed production in Malaysia to be 1.2 million metric tons [11,12]. The rubber seed oil has higher free fatty acid contents as described by the Ramdhas et al[13].

Rubber seed oil has the potential to be used as biodiesel feedstock for the biodiesel production.Parametric study of the effect of process variables on acid esterification and base transesterification of rubber seed oil was done using the Design Expert 8.0 software. The RSM (Response Surface Methodology) that includes a statistical and a mathematical tool was used to analyze and optimize the reaction parameters. The experimental design employed CCD (Central Composite Design) which is an effective, efficient and economical way of experimental techniques.

2. Experimental Procedure:

2.1 Materials and Chemicals:

The rubber seed oil was purchased from Kinetics Chemical(M) Sdn Bhd Malaysia. All the other chemicals and reagents such as anhydrous methanol (99.8%), sulfuric acid (98%) potassium hydroxide (99%) were purchased from Merck Chemicals. The FAME analytical standard was purchased from Sigma Chemicals(USA).

2.2. Rubber Seed Oil Analysis:

The basic oil analysis such as acid value, iodine value, saponification value and peroxide value were performed on the crude rubber seed oil and also treated rubber seed oil by following the AOCS method [14]

2.3 Acid Esterification :

Acid esterification is a chemical reaction in which triglycerides (oil) react with lower alcohol such as methanol in the presence of an acid catalyst and reduced the high amount of free fatty acids [15]. In this study sulfuric acid (H_2SO_4) used as the acid catalyst to reduce the FFA content. The process parameters and their ranges are shown in Table 1.

All experiments were done in 250 ml round bottom three neck flask with a reflux condenser to avoid any loss of methanol. In each run 50 g of oil is heated to desired temperature. Specific amount of methanol and sulfuric acid was added and stirring was done for specific time depending on the experimental plan. After specific time the reaction was stopped and the sample was put into the separating funnel to separate the pretreated oil and excess of methanol and catalyst. The treated oil at the bottom layer was separated and collected for further base esterification process.

2.4. Base Transesterification:

The acid value of crude rubber seed oil decreased from 84 mg KOH/g to 1.64 mg KOH. The process parameters and their ranges are shown in Table 2. In each run of the transesterification process 50 g of pre-treated oil was used which is mixed with a specific amount of methanol and potassium hydroxide (KOH). Stirring was done for a specific time depending on the experimental plan. After the specified time the reaction was stopped and the sample was put into the separating funnel for separation and left for 24 hour. Two layers were formed with the upper layer consisted of methyl ester (biodiesel) and the lower layer was glycerol, methanol and other impurities. The prepared biodiesel is collected after purification and preserved for the analysis of fatty acid methyl ester (FAME) using GC FID.

2.5. Characterization of Fuel Properties

Rubber seed oil FAME was analyzed qualitatively and quantitatively by GC FID (Gas Chromatography Flame Ionization Detector). The density was measured by using the Anton Paar DMA 4500 M Density Meter in accordance with the testing method of ASTM D4052.The FTIR (Fourier Transformer Infrared Spectroscope) was performed to analyze the conversion of fatty acid into methyl esters. Viscosity is one of the most important properties of the biodiesel and the viscosity was measured by using Brookfield CAP 200+ following ASTM D445 method at constant temperature of 40°C. Cloud point (D2500), Pour point (D97), Cold Filter Plugging Point (CFPP) (D6371), Sulfur content (D4294), Higher heating value (D4868), Acid value (D664) and Cetane number (D613), were analyzed by referring to the ASTM methods. The Oxidative stability was analyzed following EN 14114.

3. Results and Discussion:

3.1. Rubber Seed Oil Analysis:

The analysis was performed on crude rubber seed oil and treated rubber seed oil. The results were given in the Table 3.

The acid value, saponification value and peroxide values reduced after acid treatment but no significant changed in iodine value was observed. Acid treatment did not affect the degree of unsaturation of RSO [16].

3.2. Acid Esterification:

The acid esterification was performed following the design described in Table 4. The response factor which is acid value was calculated by using the AOCS method Cd 3d-63. ANOVA provides the degree of significant of model equation resulted from the optimized approached.

Perturbation plot compared all the factors influenced in a single point. The steepest curve shows the most influencing variable. The plot in figure 1 shows that factor B (Catalyst amount) is the most influencing factor towards the response value is the acid value. The second most influencing factor is A Alcohol to oil ratio.

Figure 2 shows the comparison between the actual values calculated after experimentation and the predicted values calculated by the model. From the plot it is clear that most of the points are near to the straight line, which indicates that the predicted value are close to the experimental values.

3.3. Effect of variables on acid esterification:

The reduction of acid value is significant to avoid saponifiaction reaction from occurring in subsequent transesterification process. The reduction of acid value at this stage depends on four factors, alcohol to oil ratio, catalyst amount, reaction time and reaction temperature. Catalyst amount and alcohol to oil ratios were found to be the most influencing variables as compared to time and temperature. The FFA reduction increases by increasing the amount of catalyst followed by the methanol ratio. The temperature and the reaction time both are least effective [6].

Figure 3 (a & b) clearly show that by increasing the amount of catalyst and alcohol to oil ratio the FFA decreases and figure 4 (a & b) show the trend of temperature and time obtained is similar to that reported by Ramadhas et al [13].

3.4. ANOVA Analysis :

The data obtained from the independent variables were statistically analyzed by ANOVA in order to confirm the suitability and significance of the output response variable. Table 5 shows the ANOVA for acid esterification. P-value must be less than 0.05 for the variables to have significant effect on the response values. The model F-value represents the reliability of the model and the variance of the output response. The higher the F-value for the specific

independent process variables the highest will be the effect of that variable. The catalyst amount is the most influencing process variable in the output response as compared to other three variables. The regression analysis fitted the output response with the input process variables.

The second order polynomial model equations in terms of coded and actual factors are the results of regression analysis. The second order model equations in terms of coded factors are shown below.

The regression coefficient fitted the model with respect to the actual versus predicted terms. In acid esterification the R- squared value is 0.9404, which shows that actual terms are 94.04% equal to the predicated terms.

3.5. Base Transesterification:

The base transesterification reaction was performed following the experimental design shown in Table 6. The FAME yield was calculated by using the EN 14203 method.

In this case the most influencing factor is A (Alcohol to oil ratio). In both acid esterificsation and transesterification the temperature and reaction time are least significant factors that is influenced the output response.

Figure 6 describes the trend between predicted versus experimental values. All the points are near the straight line. Thus show that software prediction confers with the experimental results obtained.

3.6. Effect of variables on transesterification:

In transesterification four variables were used for parametric study. Stoichiometrically one mole of oil needs three moles of alcohol for esterification but it was observed that excess amount of alcohol was needed to shift the reaction to forward direction. In transesterification

the most influencing factor is alcohol to oil ratio. By increasing the amount of alcohol the ester yield was increased gradually up to a certain limit. Beyond this amount of alcohol has no effect on the ester yield and higher amount of methanol ratio may hinder the glycerol separation. Similar trend was reported by Rashid et al [7].

3.7. ANOVA Analysis for Transesterification:

Table 6 shows the ANOVA analysis results for the base transesterification. The P-value of 0.0242 shows that the model is significant. Factor A (alcohol to oil ratio) has the highest F-value, thus represents the most influencing factor as compared to the other three factors.

The second order polynomial equations in terms of coded and actual factors are the results of regression analysis. The second order model equation in terms of coded factors is shown below

In this case the R- squared value is 0.9261, which shows that the actual term which is 92.61% is near to the predicated terms.

3.8. Comparison of Optimum Operating Conditions with Previous studies:

Table 8 summarized the optimum conditions of present work in comparison to previous researcher's findings.

In a nut shell, current work resulted in better conversation of FAME at optimum reaction conditions with lower amount of catalyst and less reaction time.

3.9. FTIR Analysis of RSO FAME:

The RSO FAME was also characterized by using the FTIR and the spectrum shows that the characteristic bands of the RSO FAME is between the region of 3009 – 2855 cm^{-1} which shows the symmetric and asymmetric stretching and vibration of the methyl group ($-CH_3$).

The stretching of the carbonyl group (-C=O) are in the region of 1750 – 1735 cm^{-1}, and symmetric stretching vibration of (C-O-C) in the region of 1246 – 1170 cm^{-1}. The hydrocarbon chain rocking vibration is observed in the region of 912 – 723 cm^{-1} . These results reflect the conversion of fatty acid to fatty acid methyl esters [20].

3.10. Comparison of Fatty ester profile of RSOME with SME, JME, PME, CME.

The fatty acid profile of the rubber seed oil biodiesel compare with others common feedstock used for biodiesel production such as soybean oil methyl esters, jatrohpa oil methyl esters, palm oil methyl esters and corn oil methyl ester. The rubbers seed oil fatty acid profile almost same like the soybean and corn. Both have higher amount of the unsaturated fatty acid as compared to the saturated. Due to this the cold flow properties of the SME and CME better than the PME and JME.

3.11. Fuel Properties:

The fuel properties of RSO FAME were analyzed by using the ASTM D6751 and EN 14214 standard methods. The viscosity of RSO biodiesel is within the standard thus no further modification was needed meet the standard values for the diesel engine if RSO biodiesel is used [22]. The flash point of RSO methyl ester was higher than the diesel and it is safer to be store compared to that of diesel. By blending RSO biodiesel with diesel the flash point increases and thus improved [10].The low temperature properties of RSO FAME are considerably good because unsaturated fatty acid present is higher compared to saturated fatty acids. Oxidation is one of the most important fuel property that influenced the storage and usage efficiency. Oxidative stability (OS) is related to fatty acid composition. Higher amount of saturated fatty acid have better OS as compared to higher amount of unsaturated fatty acid. RSO FAME has poor OS as compared to palm and jatropha but meets the minimum limits of EN and ASTM standards [23].

3.12. Comparison of fuel properties with previous studies:

The cetane number is a key signifier of the fuel ignition. The cetane number in the present study was found to be 54, which significantly shows more precise results compared to previous researchers work [13, 19].On the other side, Jolius et. al (2013) reported the cetane number was better than present work [17]. The oxidation stability and the CFPP of the present study show affirmative results than earlier reported studies. The oxidation stability was 8.54h which is highest among all the reported studies on the rubber seed oil biodiesel [19].The flash point of the biodiesel is the most important from storage point of view, because the higher the FP thus lesser the chances of blazing.

3.13. Kinematic Viscosity:

The kinematic viscosity is the basic property to select biodiesel as an alternative fuel instead of pure vegetable oil. Viscosity is directly depends on the nature of the fatty acids, the degree of saturation and un-saturation. The number of unsaturated fatty acids are higher then the viscosity lower and when the number of saturated fatty acid higher the viscosity should be higher. Kinematic viscosity of the rubber seed oil in the study was determined 3.89 mm^2 s^{-1}. The kinematic viscosity in this study meets both standards ASTM D6751 & EN14214. In this study the kinematic viscosity is lower than that reported studies on rubber seed oil based biodiesel [24, 25].

3.14. Cold Flow Properties:

The cold flow properties of biodiesel is generally determined by the three general parameters, cloud point, pour point and cold filter plugging point. The cold flow properties related to the composition of the fatty acid, chain length of carbon, degree of saturation and un-saturation and orientation of double bounds. By increasing the number of carbon atoms of fatty acids the melting point increased for an example the melting point of C12 is 5 °C and C18 is 39 °C. Reverse case was observed, the melting point decreases by increasing the unsaturation for an example melting point of C18:0 is 39°C, C18:1 is -20°C, C18:2 is -35°C and melting point

of C18:3 is -52°C [26]. RSO FAME shows the pour point of -2°C, cloud point of 3.2°C and CFPP of 0°C. The results of cold flow properties are better due to higher un-saturated fatty acids concentration.

3.15. Oxidative Stability:

The oxidation stability of RSO FAME was determined by using the Rancimate method described in EN 14112. The oxidation stability is the most important factor with respect to storage and performance of fuel. The increase in viscosity, gumming and deposition of unwanted particle during the storage results in poor oxidation stability of biodiesel [26]. The oxidation stability of RSO FAME was 8.54 h for this work and is higher than all the previous studies on the rubber seed oil biodiesel. This value meets both the standard values of ASTM D6751 and EN 14214.

4. Conclusion:

The response surface methodology was used for the parametric study on the reduction of FFA and production of biodiesel from rubber seed oil. Four different types of variables were studied. Catalyst concentration and alcohol to oil ratio were the most influencing factors in FFA reduction but for base transesterification the observed trend is reversed. Reaction time in both cases has the least effect. The maximum conversion of methyl esters (biodiesel) was 96.8% at reaction variables of 6:1 alcohol to oil ratio, 1% catalyst concentration, 55 $^{\circ}C$ reaction temperature and 67.5 minute reaction time. All the properties of biodiesel were within the range of standards including the viscosity, flash point, iodine value, ester content and caloric value. Present study clearly proves that crude rubber seed oil (non-edible oil) is a potential feedstock for the production of biodiesel.

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Table 1 Process parameter for Acid Esterification

Process Parameters	-2	-1	0	+1	+2
Oil to Methanol (molar ratio)	8.3	10	12.5	15	16.7
Catalyst Loading (wt%)	3.3	5	7.5	10	11.7
Temperature (°C)	38.2	45	55	65	71.8
Time (minutes)	43.1	55	72.5	90	101.9

Table 2 Process parameter for base transesterification.

Process Parameters	-2	-1	0	+1	+2
Oil to Methanol (molar ratio)	0.95	3	6	9	11.05
Catalyst Loading (wt%)	0.16	0.5	1	1.5	1.84
Temperature (°C)	38.16	45	55	65	71.8
Time (minutes)	29.6	45	67.5	90	105.4

Table 3: Rubber seed oil Analysis Results.

Analysis	Units	Before Treatment	After Treatment
Acid Value	mg KOH/g oil	84	1.8
Peroxide Value	mg/g	1.6	0.7
Iodine Value	I_2 /g oil	146	146
Saponification Value	meq/kg	194	186

Table4: Experimental design for acid esterification.

Experimental Run	Alcohol to Oil Molar ratio	Catalyst Amount (wt%)	Time (min)	Temperature (^{o}C)	Response (FFA%)	Predicted Response (Acid value)
1	8.3	7.5	72.5	55	3.12	3.08
2	12.5	7.5	72.5	38.2	1.16	1.14
3	16.7	7.5	72.5	55	0.85	0.84
4	15	5	90	65	0.91	0.90
5	12.5	7.5	72.5	55	1.12	1.13
6	12.5	7.5	72.5	55	1.12	1.12
7	15	10	55	45	1.23	1.22
8	15	5	55	65	1.09	1.08
9	10	10	90	65	1.21	1.20
10	10	10	10	55	1.99	1.97
11	15	10	90	45	0.82	0.81
12	10	5	90	45	2.58	2.57
13	12.5	7.5	72.5	55	1.11	1.10
14	12.5	7.5	101.9	55	0.86	0.85
15	12.5	7.5	43.1	55	1.60	1.62
16	12.5	7.5	72.5	71.8	1.55	1.53
17	12.5	7.5	72.5	55	1.11	1.09
18	12.5	11.7	72.5	55	0.89	0.88
19	12.5	3.3	72.5	55	4.96	4.95
20	10	5	55	45	2.20	2.19
21	12.5	7.5	72.5	55	1.12	1.10

Table 5: ANOVA analysis of acid esterification

Source	Sum of Squares	DF	Mean Square	F value	P value
Model	18.46	14	1.32	6.76	0.0136 significant
A (Alcohol)	2.58	1	2.58	13.21	0.0109
B (Catalyst)	8.28	1	8.38	42.45	0.0006
C (Time)	0.37	1	0.37	1.87	0.2201
D(Temperature)	0.076	1	0.076	0.39	0.5554
AB	0.34	1	0.34	1.74	0.2357
AC	4.513E-003	1	4.513E-003	0.023	0.8841
AD	3.44	1	3.44	17.63	0.0057
BC	0.24	1	0.24	1.24	0.3085
BD	0.11	1	0.11	0.57	0.4778
CD	0.35	1	0.11	0.55	0.4848
A^2	3.53	1	0.35	1.80	0.2281
B^2	0.19	1	3.53	18.07	0.0054
C^2	0.072	1	0.19	0.99	0.3586
D^2	1.17	1	0.072	0.37	0.5660
Residual	1.17	6	0.20		
Lack of Fit	8.000E-005	2	0.59	29263.09	<0.0001 significant
Pure Error	18.46	4	1.32	6.76	
$R^2 = 0.9404$	$R^2_{adj} = 0.8012$		Adequate precision= 11.010		

Tabe 6: Experimental design and response of FAME conversion.

Run	Methanol/oil	Catalyst	Temperature	Time	Palmatic	Stearic	Oleic	Linoleic	Linolenic	Total	Response Yield(%)	Predicted Yield(%)
1	6	1	55	67.5	9.84	9.89	24.74	35.18	16.76	96.41	81.40%	81.2
2	6	1.84	55	67.5	8.93	8.55	23.63	34.55	7.97	83.64	40.10%	40.11
3	6	1	55	67.5	9.89	9.9	24.89	35	16.78	96.46	83.02%	83.01
4	0.95	1	55	67.5	7.2	6.6	12.8	18.87	8.83	54.38	25%	24.9
5	6	1	71.8	67.5	8.95	8.3	23.5	34.57	16.79	92.11	62.08%	62.06
6	9	1.5	45	45	8.82	8.38	23.42	34.55	16.78	92.08	72.10%	72.09
7	3	1.5	45	90	9.14	8.47	24.08	35.69	17.14	94.79	64.40%	64.3
8	9	1.5	65	45	8.72	8.14	22.98	33.88	16.42	90.14	73%	72.9
9	3	0.5	45	45	9.23	8.62	24.04	35.08	16.83	93.8	85.86%	85.87
10	6	0.16	55	67.5	8.83	8.2	23.13	34.12	16.59	90.87	60%	60.1
11	3	1.5	65	90	8.86	8.35	23.85	35.49	16.48	93.03	75%	74.98
12	6	1	55	67.5	9.08	9.57	24.95	35.56	16.98	96.14	81.50%	81.49
13	6	1	55	29.6	7.21	6.61	18.69	27.72	13.47	73.7	67.32%	67.30
14	3	0.50	65	45	8.2	8.08	22.16	35.19	15.95	89.58	87.30%	87.20
15	11.05	1	55	67.5	8.05	7.42	20.93	30.97	10.17	77.54	88.50%	88.40
16	6	1	55	67.5	9.83	9.9	24.87	34.93	16.81	96.34	83.28%	83.20
17	9	0.50	45	90	8.98	8.47	23.6	34.73	16.81	92.59	81%	80.90
18	9	0.50	65	90	9.67	9.16	25.51	37.59	18.23	92.06	80.56%	80.54
19	6	1	38.18	67.5	6.47	5.57	16.69	24.76	11.92	65.59	81.38%	81.35
20	6	1	55	67.5	9.9	9.89	24.87	35.17	17.01	96.84	81.80%	81.78
21	6	1	55	105.4	9.17	8.61	23.99	35.28	17.08	94.13	82.2%	82.10

Table 7: ANOVA analysis of transesterification.

Source	Sum of Squares	DF	Mean Square	F value	P value
Model	4380.74	14	312.9	5.37	5.37 significant
A (Alcohol)	2178.00	1	2178.00	37.38	37.38
B (Catalyst)	12.50	1	12.50	0.21	0.21
C (Time)	15.39	1	15.39	0.26	0.26
D(Temperature)	1.13	1	1.13	0.019	0.019
AB	16.01	1	16.01	0.27	0.27
AC	2.61	1	2.61	0.045	0.045
AD	91.59	1	91.59	1.57	1.57
BC	94.46	1	94.46	1.62	1.62
BD	1426.85	1	1426.85	24.49	24.49
CD	76.20	1	76.20	1.31	1.31
A^2	522.23	1	522.23	8.96	8.96
B^2	553.93	1	553.93	9.51	9.51
C^2	22.49	1	22.49	0.39	0.39
D^2	394.37	1	394.37	6.77	6.77
Residual	349.62	6	58.27		
Lack of Fit	346.49	2	173.25	221.49	< 0.0001 significant
Pure Error	4380.74	4	0.78		
$R^2 = 0.9261$	$R^2_{adj}=0.7536$		Ad. precision= 10.230		

Table 8: Comparasion of process variables with previous studies.

Parameters	Ramadhas et al.[12]	Jolius Gimbun et al[17]	M.Morshed et al[18]	R.Yang et al[19]	This work
Alcohol/oil ratio	6:1	4:1	5:1	6:1	6:1
Catalyst	0.5	6	5	-	1
Temperature	45	65	-	60	55
Time	30	240	100	-	67.5

Table 9: Comparasion of fatty ester profile of RSOME with SME, JME,PME and CME.

FAME (wt %)	RSOME	SME[21]	JME [21]	CME [21]	PME [21]
Palmatic (C16:0)	9.89	11	12.15	11.67	44
Stearic (C18:0)	9.9	4	16.80	1.85	4
Oleic (C18:1)	24.89	22	13	25.16	40
Lionleic (C18:2)	35	53	49.75	60.60	10
Linolenic (C18:3)	16.78	8	-	0.48	-
Arachidi (C20:0)	-	2	5	0.24	2
Eicosenic (20:1)	-	-	2	-	-

Table 10: Fuel Properties of RSO Biodiesel.

Property	**RSOFAME**	**ASTM D6751**	**EN14214**
Density (25 $^{\circ}$C) kg m^{-3}	885	N/A	860-900
Viscosity (mm^2 s^{-1}; 40 $^{\circ}$C)	3.89 (Check ref.)	1.9 – 6.0	3.5 – 5
Cetane Number	54	47 min	51 min
Oxidative Stability (h)	8.54	3 min	6 min
Cloud Point ($^{\circ}$C)	3.2	-	-
Pour Point ($^{\circ}$C)	-2	-	-
Cold Filter Plugging Point ($^{\circ}$C)	0s	-	-
Flash Point ($^{\circ}$C)	152	93	120
Calorific Value (MJ kg^{-1})	39.70	-	-
Sulfur Content (%)	0.02	0.05 max	-
Free Glycerine (%)	0.02	0.02 max	0.02 max
Total Glycerine (%)	0.35	0.240	0.250
Acid Value (mg KOH g^{-1})	0.42	0.50 max	0.50 max
Ester Content	96.8	N/A	96.6

Table 11: Comparasion of fuel properties with previous studies of rubber seed oil biodiesel.

Properties	Ramadhas et al.	Jolius Gimbun et al	M.Morshed et al	R.Yang et al	This work
Cetane Number	43	66.2	-	49.6	54
Oxidative Stability (h)	-	-	-	6.5	8.54
Cloud Point ($^{\circ}C$)	4	-	3	-	3.2
Pour Point ($^{\circ}C$)	-8	-	-5	-	-2
CFPP ($^{\circ}C$)	-	-	-	-1	-2
Flash Point ($^{\circ}C$)	130	154.6	120	150	152
Calorific Value (MJ kg^{-1})	36.5	39.37	32.6	-	39.70
Ester content	-	96.9	98	-	96.8
Viscosity (mm^2 s^{-1}; 40 $^{\circ}C$)	5.81	4.65	4.5	4.059	3.89

Figure(s)

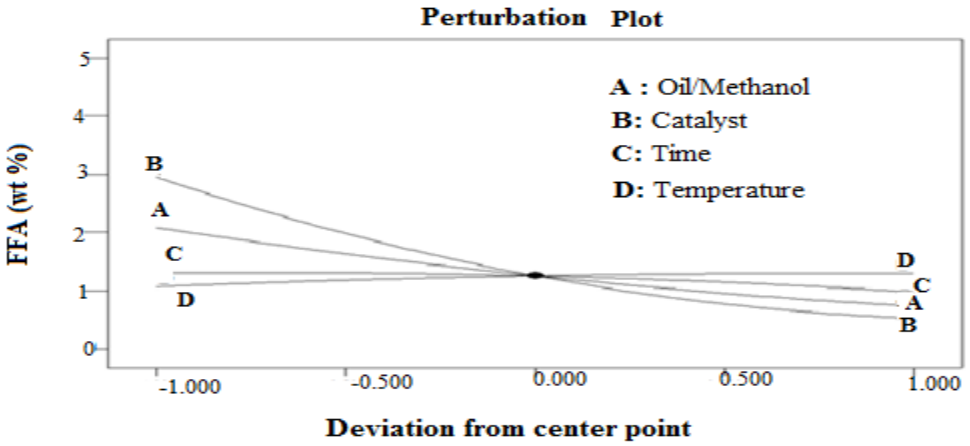

Fig. 1

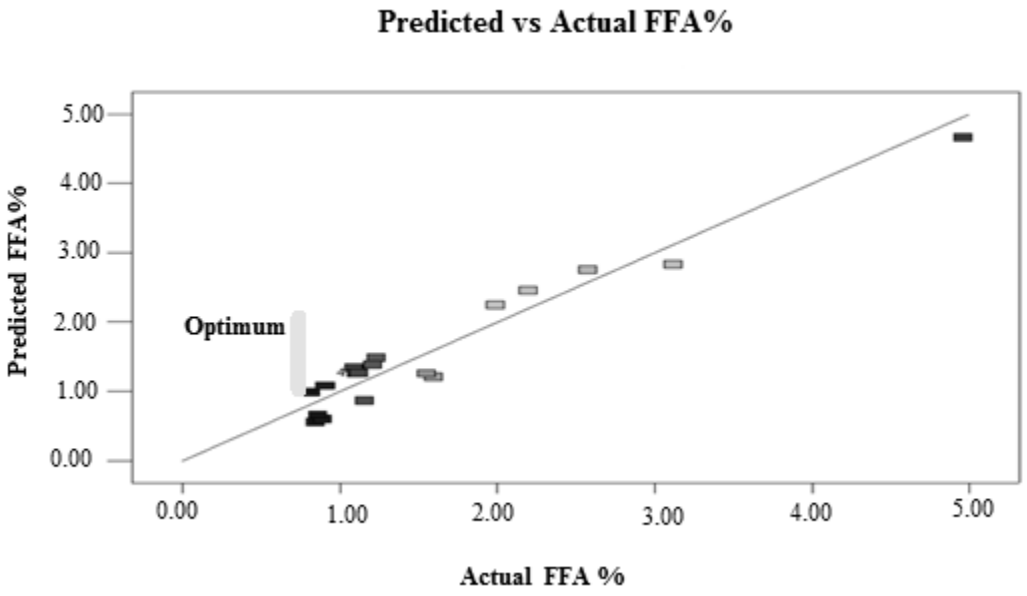
Predicted vs Actual FFA%
Predicted FFA%
Actual FFA %
Optimum
5.00
4.00
3.00
2.00
1.00
0.00
0.00
1.00
2.00
3.00
4.00
5.00

Fig. 2

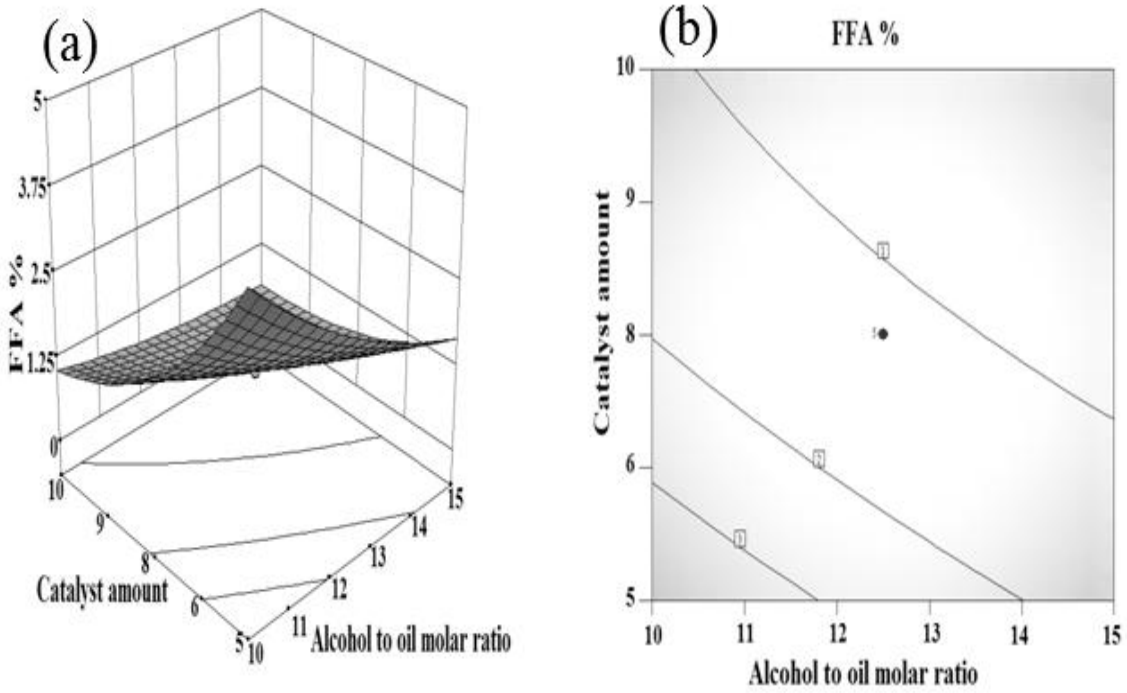
(a)
FFA %
5
3.75
2.5
1.25
0
10
9
8
6
5
Catalyst amount
10
11
12
13
14
15
Alcohol to oil molar ratio
(b)
FFA %
10
9
8
6
5
Catalyst amount
10
11
12
13
14
15
Alcohol to oil molar ratio

Fig. 3

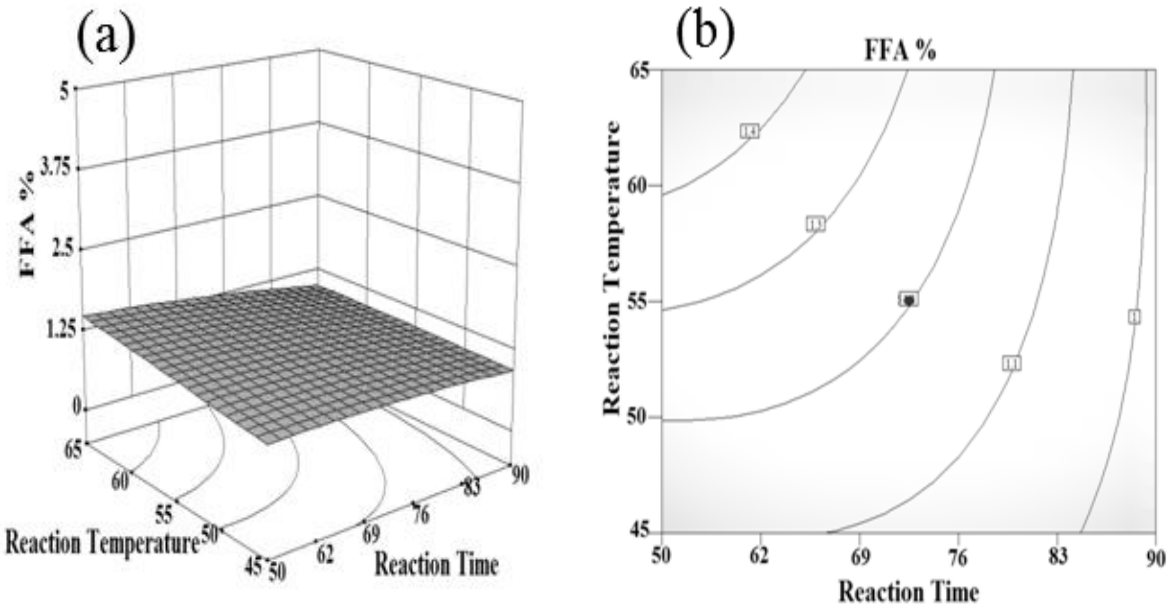

Fig. 4. show the combined effect of reaction time and reaction temperature (a) show th three dimisi

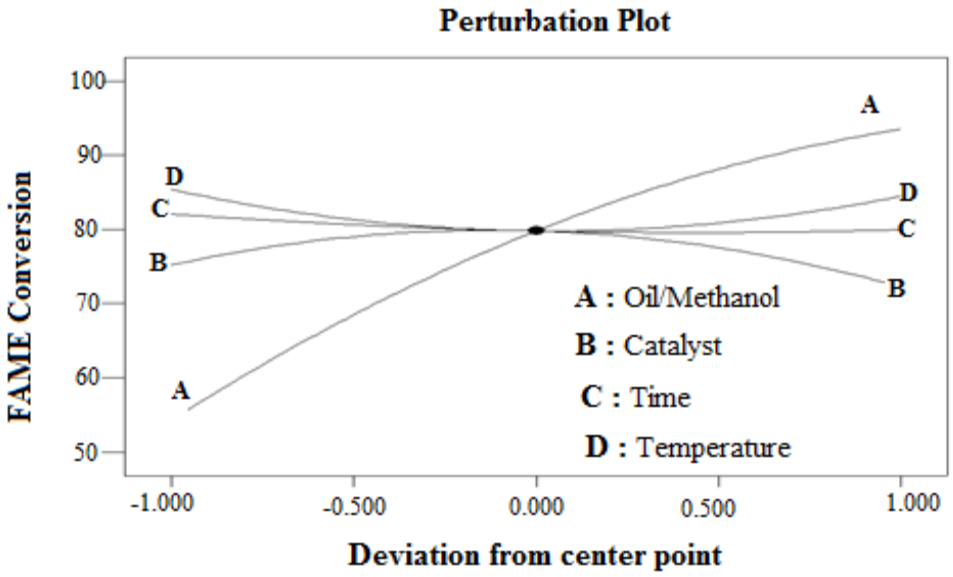
Perturbation Plot
FAME Conversion
100
90
80
70
60
50
-1.000
-0.500
0.000
0.500
1.000
Deviation from center point
A
D
C
B
A : Oil/Methanol
B : Catalyst
C : Time
D : Temperature

Fig. 5

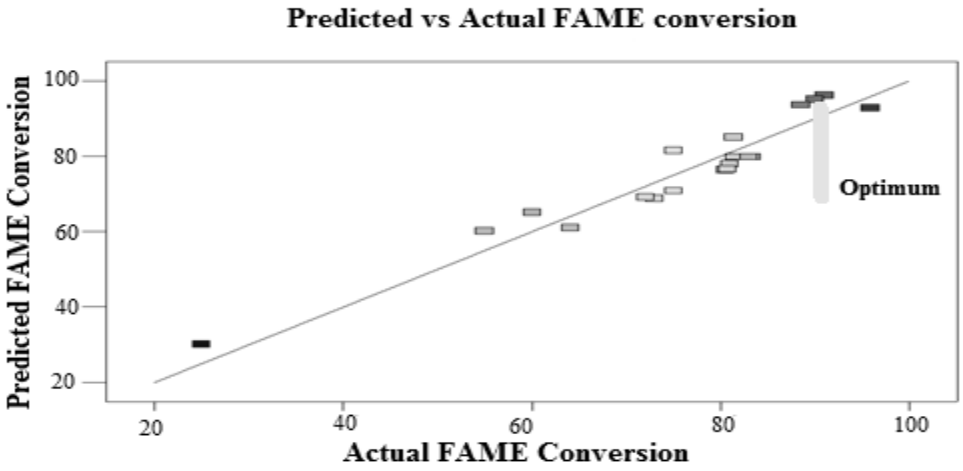
Predicted vs Actual FAME conversion
Predicted FAME Conversion
100
80
60
40
20
20
40
60
80
100
Actual FAME Conversion
Optimum

Fig. 6

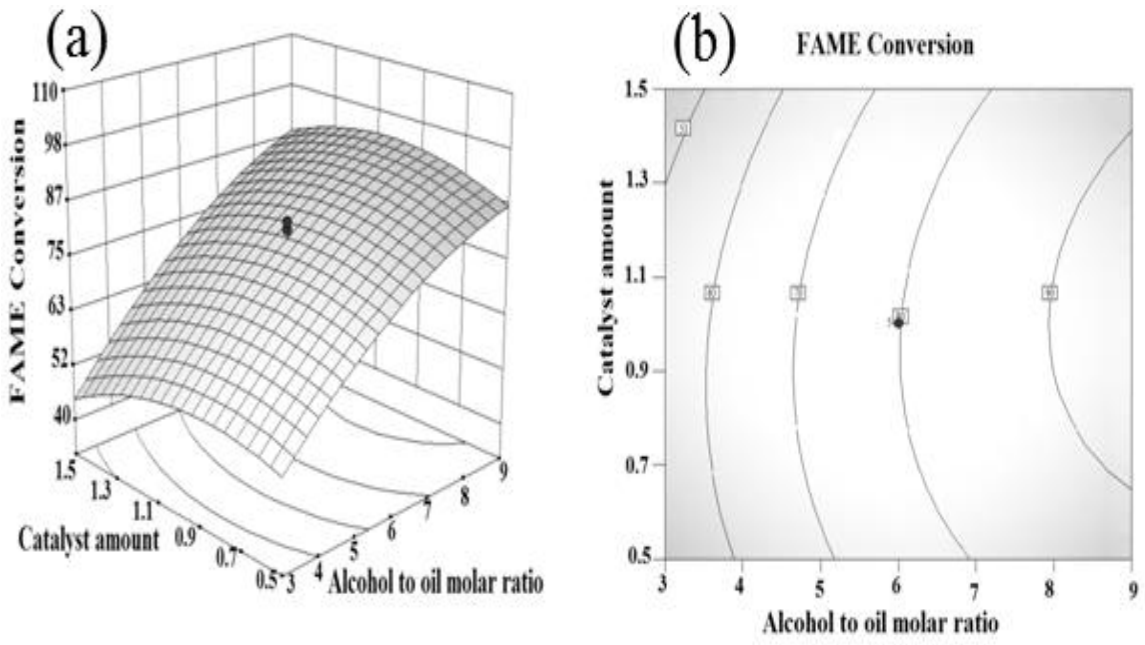
(a)
FAME Conversion
110
98
87
75
63
52
40
1.5
1.3
1.1
0.9
0.7
0.5
Catalyst amount
3
4
5
6
7
8
9
Alcohol to oil molar ratio
(b)
FAME Conversion
Catalyst amount
1.5
1.3
1.1
0.9
0.7
0.5
3
4
5
6
7
8
9
Alcohol to oil molar ratio

Fig. 7

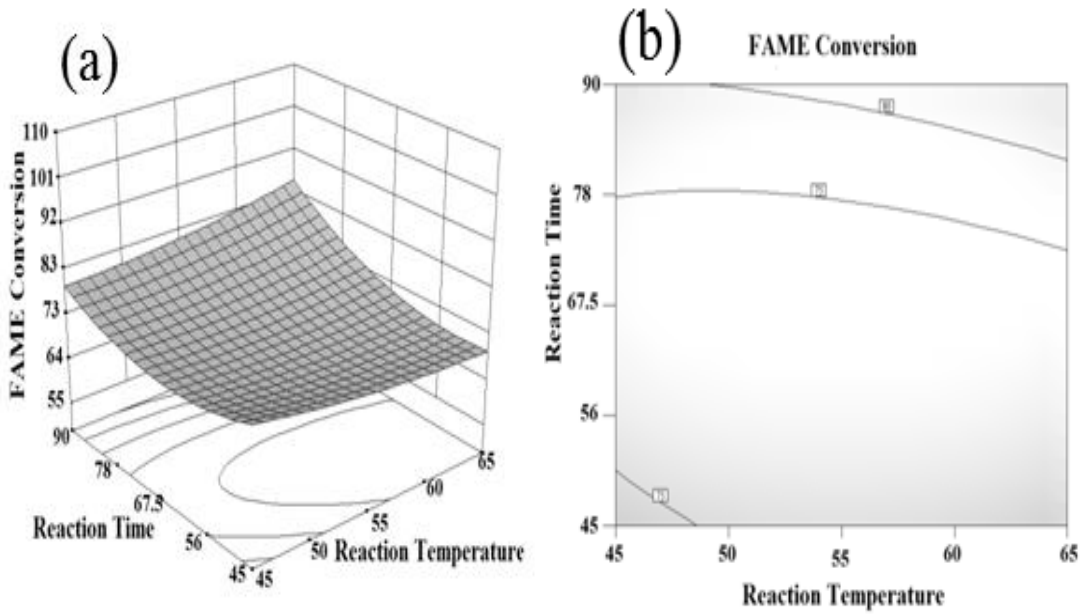
(a)
FAME Conversion
110
101
92
83
73
64
55
90
78
67.5
56
45
45
50
55
60
65
Reaction Time
Reaction Temperature
(b)
FAME Conversion
90
78
67.5
56
45
45
50
55
60
65
Reaction Time
Reaction Temperature

Fig. 8

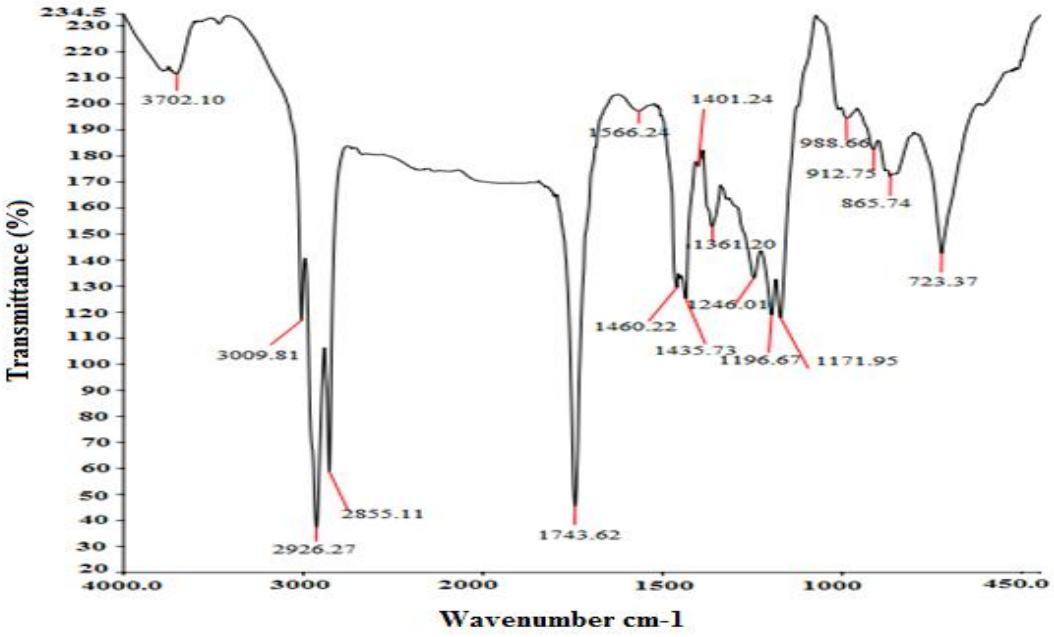
Transmittance (%)
Wavenumber cm-1
3702.10
3009.81
2926.27
2855.11
1743.62
1566.24
1401.24
1460.22
1435.73
1361.20
1246.01
1196.67
1171.95
988.66
912.75
865.74
723.37

Fig. 9