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Characterization and Experimental Analysis of Biodiesel Extracted from Sclerocarya Birrea (Marula) Fruit Using Catalyst

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Abstract

The investigation of indigenous resources as alternative energy is the reasonable solution for the diminishing of naturally existing materials for production of fuel from crude oil, environmental concern. Many researchers have been involved in producing a low-price fuel from naturally existing raw materials. Thus, in this specific work biodiesel production was examined from Marula seed. The seeds were collected from Arba Minch NechSarNationalPark and the seeds proximate analysis has been conducted to determine moisture content, ash content, crude fiber content and fat contents. The result found was 5.90%, 4.27%, 11.84% and 47.66% respectively. The extraction of the oil was performed by solvent method using n-hexane as solvent through Soxhlet apparatus and fixing the extraction time. This study aimed to extract a biodiesel from sclerocarya birrea by examining the physio-chemical properties, and the biodiesel production was optimized for pre-determined parameters such as Catalyst Concentration (CC), Reaction Temperature (RT) and Methanol to Oil Ratio (MOR) by implementing Design Expert Software (DES).The optimum conversion efficiency of marula oil to Fatty Acid Methyl Ether (FAME) was 93.45% at optimal condition of 9:1 methanol to oil ratio for 1.75%of catalyst loading at 60^{o}C of reaction temperature. The properties of sclerocaryabirrea which were determined exist in the recommended standards. The oil content of the seed was found 41.57%. The values of the physiochemical properties of the oil were viscosity 92.8mpas, specific density 0.923, acid value 7.51, Saponification value 229miligrams and free fatty acid 3.8% and the biodiesel were characterized as of its calorific value (CV) which was 42.56 MJ/Kg, viscosity of 11.3mpas, iodine value of 115 I_2/gm, and flash point of 235^{o}C.In this research work, it was concluded that Sclerocaryabirrea can be a possible input for biodiesel production which in turn minimize the dependency on fossil fuels.

Key words: Biodiesel, free fatty acid, transesterification and Proximate analysis

1 INTRODUCTION

In recent days, the instability of petroleum price and diminishing of input material for these fuels initiated scholars searching other resources like non-edible plant seed oils as a substitute for crude petroleum .Today biodiesel is the essential alternative diesel fuel in the total production of fuel in European union which accounts around 82% (Bozbas,2008).

For diesel substitution biodiesel is a good option as an alternative energy particularly catalyst involved in the production of biofuel (Mata and martins, 2015)

In fact, in Ethiopia, due to lack of appropriate technology and shortage of detail research finding information on biodiesel, fossil fuels are the important source of energy for domestic and industrial usage. The utilization of petroleum fuels can be the reasons for contamination and results in diminishing of economic growth (Laherrere, 2005). Therefore, fossil fuels became one of the issues which can cause conflict in developing countries due to high price increment every year. The alternative energy source gets high attention particularly for transportation fuel and increasing interest for research in this area leads to be developed independent energy sources.

In acquiring a biofuel with a good quality, searching for recent feed stocks and adaptation measuring technologies have a paramount role (Geissler, 2008).There are a lot of resources which are still not utilized for further studies for biodiesel production (Winayanuwattikun et al. 2008).

These indicates more than 95% of biodiesel that can be produced comes from edible feedstocks which contains oil because the properties of these oils are comfortable in the production of biodiesel in order to replace fuel from the diesel (Gui et al. 2008).

However, the use of edible crops for biodiesel in developing countries like Ethiopia can cause competition with the edible oil market (Kansedo et al. 2009). In order to overcome the above aforementioned challenges, it is timely to search non-edible oil sources which are not suitable for human consumption. Since the cost of raw materials accounts about 60–80% of the total cost of biodiesel production, choosing a right feedstock is very important (Guiet al. 2009; Singh and Singh, 2009).

Ethiopia is rich in plant biodiversity which can have potential for utilization of biodiesel; Therefore, the present study was tested the suitability of Marula tree, which is the non-edible oil trees commonly available in different parts of the country for the production of biodiesel. Even though, marula tree is widely growing fruit in many parts of the country (Amhara, Southern region, Oromiya and Tigray), there is no research which reveals the suitability of Marula oil as biodiesel

2 Materials and Methods

Study sample

Raw materials, ripened fresh fruits, of Marula trees (Sclerocaryabirrea) were collected from *Arba Minch, NechSar National Park* and transported to laboratory (School of Chemical and Bioengineering), Addis Ababa Institute of Technology, Addis Ababa University for further analysis. The chemicals and Equipments utilized during the Experiment is indicated in (Table 1).

Table 1 Equipment and chemicals utilized

Equipment used	Drying oven,
	Weighing balance,
	a Soxhlet apparatus,
	Round flask

	Water bath
	viscometer
	condenser
	Three neck flasks
	Burette
	Dense meter
	Beaker
	Holding clamp
Chemicals used	potassium hydroxide
	Phenolphthalein
	Diethyl ether
	Ethanol
	Hydrochloric acid
	Sodium hydroxide
	Methanol

Procedures

The seed were collected from Arbaminch NechSar Park, from Southern Nation Nationalities and Peoples of Ethiopia and were transported to Addis Ababa University, Addis Ababa Institute of Technology for conducting the research. The seed of marula were prepared after the outer cover is crushed for oil Extraction from it. The solvent utilized for the extraction of the was n-hexane and after the oil is purified and degummed it was used for biodiesel production and the overall procedures of biodiesel production is briefly described in (Figure 1).

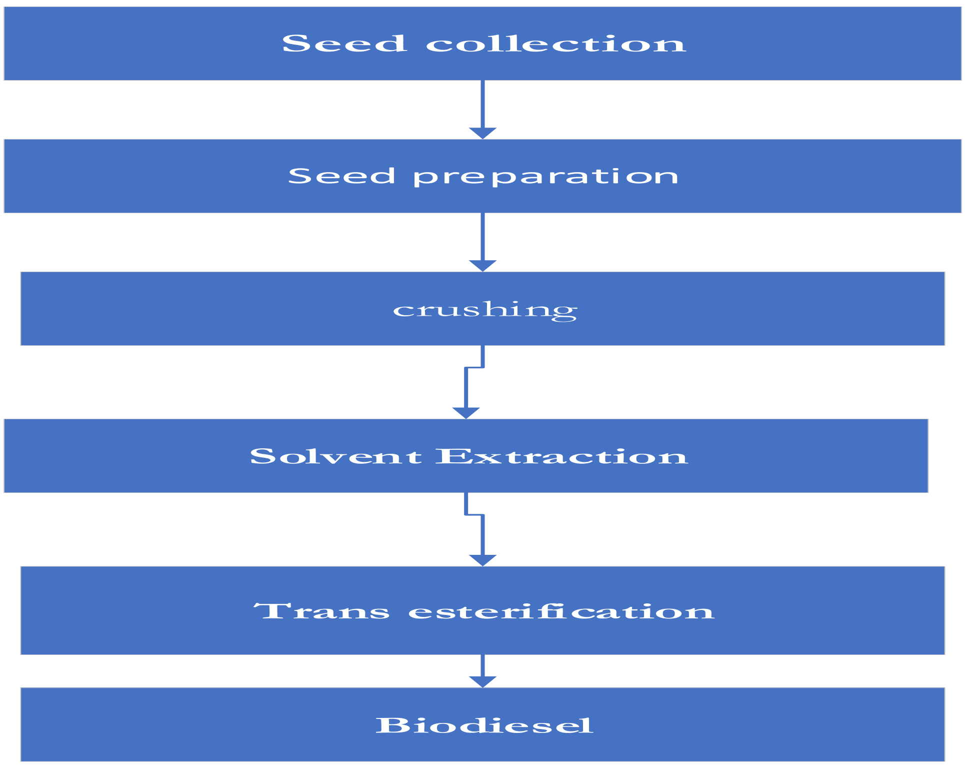

Figure 1: Flow chart of biodiesel production

Marula Seed collection and preparation

The collected seed was prepared by removing the outer coverage of the kernel, then dehulling the

Kernel and finally the endocarp of the seed were prepared.

Figure 2: Seed collection, removing the outer cover

Figure 3: Dried marula and its internal parts which contain oil

Proximate analysis and Characterization of Raw material (marula Seeds)

Physio-characteristics of marula oil were examined according to the usual procedure recommended by AOAC (1980). The oil properties including moisture content, crude fiber, fat, fixed carbon content, volatile matter and ash contents were determined.

Moisture Content Determination of Marula seed

Content of moisture of the seed was indicated by the procedure set as of (AOAC, 930.15, 2016). Marula seed should be added to the dish and the dish without marula weighed and recorded, then the dish with marula seed inserted in to the oven dry by fixing the temperature at 105 degree Celsius for consecutive seven hours and the weight of the seed measured after two hours until fixed weight obtained and the formula used for determination of moisture content is indicated in equation (1).

Moisture content, % $= \frac{w2-w3}{w2} * 100$ **---------------------------------- (1)**

Where, w2=weight prior dried w3= seeds weight when dried

Ash content of the seed

The standard procedure used in examining the ash content of marula seed was as per the method of AOAC, 923.03; 2016.Itindicates the impurities that will not burn. The presence of high ash content affects the combustion efficiency of fuels and cause slagging and clinkering. Typical ranges of ash content should be 3% to 10% (A.K.Shaha, 2011) and the variation range is extended due to the origin of solid biofuels, species, climatic condition and specific part of the selected plant. The following formula was used in determining ash content of marula seed

3 Results

3.1.1 Proximate analysis marula seed, extraction and characterization of the marula oil

Proximate analysis of the raw material of Sclerocarya birrea seed was determined and tabulated as indicated in the (Table2).

Table 2: Summary of Proximate analysis of marula seeds

Proximate analysis	Percentage	Test methods used
Moisture content	5.90%w/w	AOAC 930.15,2016
Crude fiber content	11.84%w/w	AOAC 962.09,2016
Fat content	47.66%w/w	AOAC 2003.06,2016
Ash content	4.27%w/w	AOAC 923.03,2016

Table 2 above demonstrates moisture content of 5.90%w/w which is very desirable and doesn't affect the yield of the oil production but since fat content of the seed is very high which is 47.66%w/w it has great effect during production of biodiesel, this indicates it needs removal of fat from oil before production of biodiesel by neutralizing the oil. The ash content of the seed which forms impurities is tolerable which fails in the standard 4.27%w/w.

3.2 Oil extraction of marula

The desired amount of marula oil was extracted from the collected seeds using Soxhlet apparatus. The extraction step was prepared for 92.22 g of the sample and the experiment was repeated in triplicate. The oil content (oil yield) of the dried marula seeds was determined by

(Equation 2).

3.3 Crude Oil Purification and characterization of physiochemical properties of the oil

3.3.1 Oil degumming

The extracted oil from marula seed initially heated to the temperature of seventy degree Celsius and rousing up to thousand revolutions per minute (1000 rpm) in a jacketed glass vessel linked to centrifugal machine. it was stirred for about half hour(0.5hr)following the formation of black residue inside the vessel. Positive gum of the oil was removed by water and the negative gum removed by phosphoric acid.

3.3.2 Washing of the oil and neutralization

Washing of the purified oil to remove free fatty acid present in the oil, boiled water was added on to the oil that exists in the separatory funnel and the addition of water continued until the removal of FFA was completed. At the end the oil was taken into the oven dry for drying setting its temperature at 100°C to remove the water present as indicated in the (Figure 4).

Figure 4: crude oil washing and neutralization

3.3.3 Determination of percentage of oil yield

The amount of oil can be determined after extraction of oil from the seed of marula. The oil extraction was carried out using Soxhlet apparatus and the solvent present in the oil after extraction was recovered by simple distillation using vacuum rotary evaporator and the water bath temperature was set at 65 °C, the fooling formula was used in determination of oil yield.

$$\textbf{Oil yield} = \frac{mo}{ms} \textbf{x}100\% \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad \textbf{(2)}$$

Where *mo*= weight of oil recovered after extraction
ms= weight of seed samples

Table 3: Oil yield of s.birrea

Ru n	Sampl e (g)	V (cm3)	M=s* v	Yiel d (%)	Averag e

1	92.22	38.2	35.29	38.2	41.57
	6		7		
2	92.22	40.3	37.24	40.3	
	5		8		
3	92.22	45.9	42.45	46.0	
	9		3		

The average oil content of the seed was calculated and obtained an average of 41.57%. This indicated that the marula seed has elevated oil content which can be good potential for biodiesel production with non-edible plant that could not compete with human food consumption

3.4 Characterization of physiochemical properties of marula oil

The physicochemical properties of the oil were studied to determine its potential for use as a biodiesel feedstock. The physicochemical parameters suggest the suitability of the oil for conversion into biodiesel. The content of Free Fatty Acid (FFA) of the oil was slightly high; as a result earlier reports suggested that higher FFA content lowers the biodiesel yield which may be due to formation of soap which makes the separation of biodiesel difficult. However, in the present study, the yield was reportedly increased by employing a heterogeneous catalyst. Similarly, reported the use of a heterogeneous catalyst in the preparation of biodiesel production the comparison of physiochemical properties of marula seed conducted before and in this study indicated in (Table 4).

Table 4: Physiochemical properties of marula oil

Physiochemical properties of the oil	units	Bikila	Gadisa
Average oil content	%	41.57	61.36
Time of extraction	hr	3-5	3
Volatile matter	%	84.62	-
Fixed carbon content	%	5.22	-
Acid value	mgKOH/g	7.52	3.6
Free Fatty Acid	%	3.8	1.81
Saponification value	mgKOH/g	229.015	190
Ester value	mgKOH/g	221.49	--
Kinematic viscosity	mm^2s^{-1}	0.1024	-
Density	$gcm-^3$	0.906	-
Specific gravity at 15ºC	-	0.923	0.899
Refractive index	-	-	1.467

3.5 Oil optimization using design expert

During the optimization of the oil two parameters were employed, these are time in hour and temperature in degree celisius.as indicated in the Table 3.4 the model and the two parameters were significant.

3.5.1 ANOVA analysis of oil yield

Table 5 Anova Analysis in puts

Source	Sum of Sq	df	Mean Sq	F Value	P Value	
Models	789.11	3	263.04	230.21	0.0001	significant
*A-Temperature	130.32	1	130.32	114.06	0.0001	
B-Time	61.03	1	61.03	53.41	0.0001	
AB	453.21	1	453.21	396.66	0.0001	
Residual	10.28	9	1.14			
Lackof Fit	10.28	1	10.28			
Pure Error	0.0000	8	0.0000			
Cor Total	799.39	12				

Coefficient is statistically significant at P<0.05

*Dependent variable: A-Temperature

The mean square value and the value of F illustrates the model is important and indicates significance of the model, the value of P, which is below 0.0500 demonstrates the model terms accuracy.

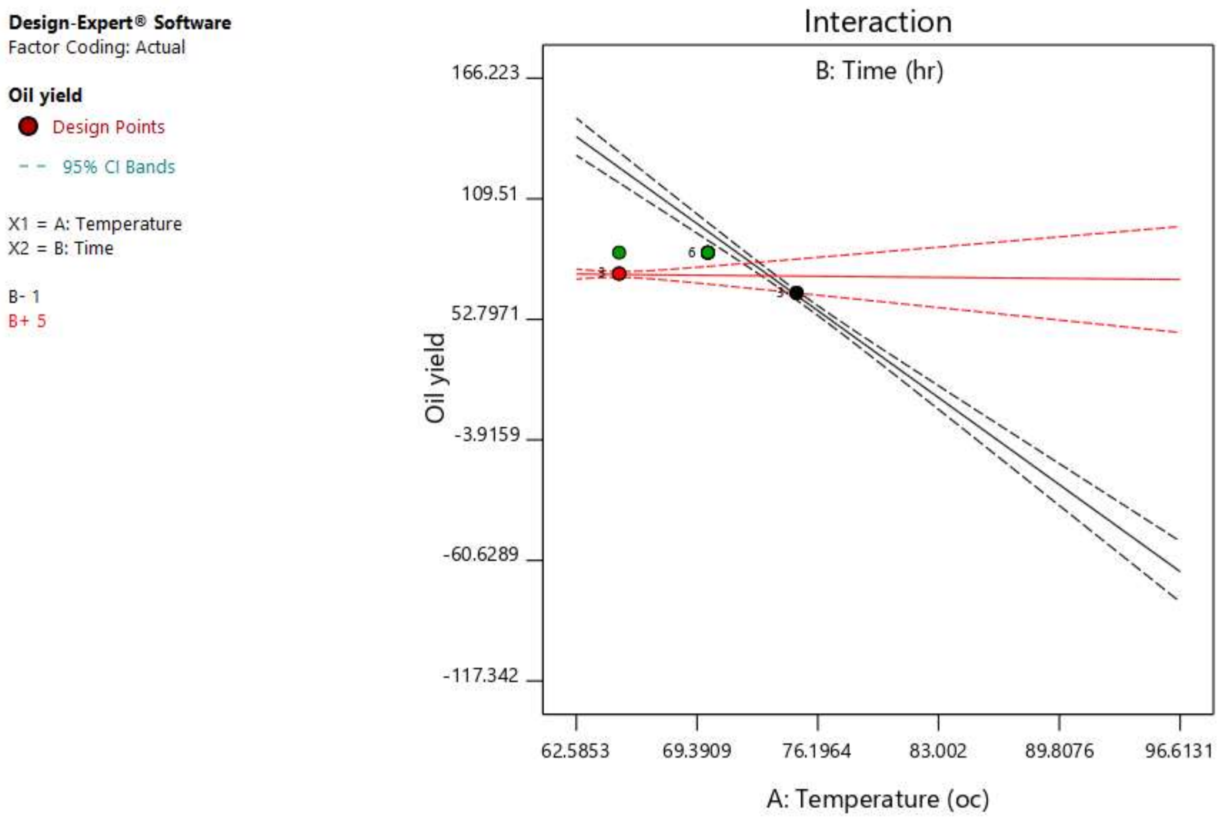

Figure 5: Interaction graph

The graph indicates the relationship between temperature, time and oil yield. The Figure illustrates that as temperature increases the product or oil yield decrease but at medium temperature with effective time the product can be increased.

The graph of the estimated results obtained using the developed correlation versus actual values forms a line of unit sloped correlation versus actual values forms a line of a unit slope, i.e. the line of a perfect fit with points corresponding to zero error between predicted values and actual values. The Figure (6) indicates the points of actual value and predicted value scattered on the line concisely which demonstrates the actual value and the predicted value are nearly close to each other. From the graph the actual value is around 85% and that of predicted value is around 86% that illustrates the values are close to each other for which the model is perfectly fit.

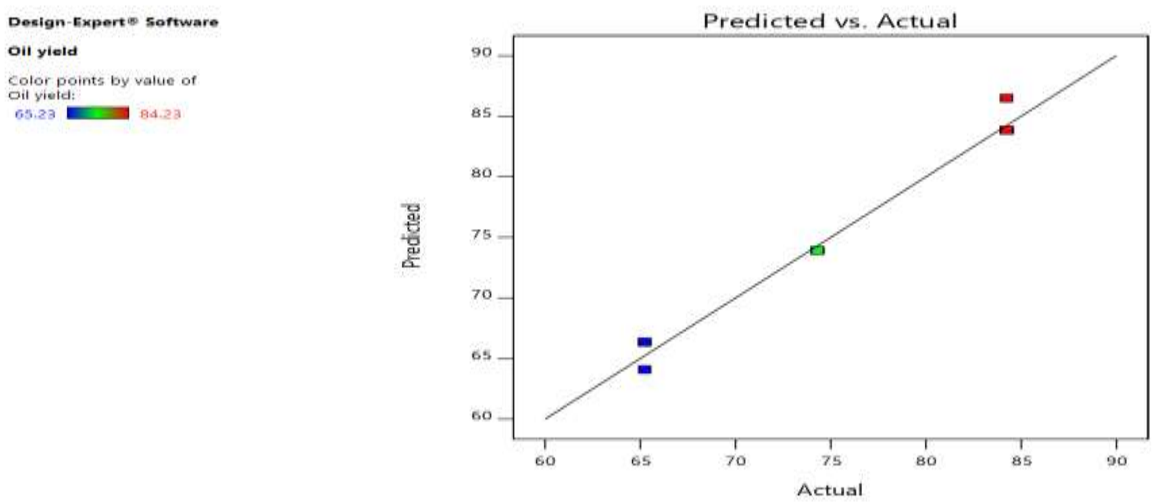

Figure 6 Predicted versus actual graph including contour line indication

3.6 Physicochemical Properties Analysis of Biodiesel (characterization) of marula

Initially checking physicochemical properties of marula oil is very important, because parameter such as free fatty acid, moisture content is highly affected next transesterification reaction and yields of biodiesel. This was the major properties of oil that help in decision making for next process to select which types of transesterification appropriate, types of catalyst and to estimate oil quality as well as oil content of given feedstock. Physicochemical properties of the samples of biodiesel were analyzed for its density, specific, calorific value gravity; viscosity, iodine value, and flash point were tabulated in (Table 6). All determinations were done three times and the average values were taken.

Table 6 Physiochemical properties of biodiesel

Physicochemical properties	units	value
Iodine value	mgI_2/gm	115.0
Specific gravity	-	19.96

Density	g/cm^3	0.915
Flash point	^oC	235.0
viscosity	mpa.s.	11.30
Heating value(calorific value)	MJ/Kg	42.55

3.7 Biodiesel optimization and graphical analysis using design Expert software

3.7.1 Process of Transesterification

The biodiesel yield was investigated mainly for the three major effect parameters namely methanol to oil ratio (MOR), Temperature (T) and Catalyst Concentration (CC). In this investigation, the reaction time and mixing intensity kept constant for all experimental runs (1hr and 500rpm) respectively (Mathiyazhagan, M, 2011). Since Soxhlet extraction oil produces high-quality oils, degumming was enough to reduce the acid values or no need for esterification process in order to reduce the acid values. Twenty five milliliters of degummed marula oil was used for each run under given constraint. The statistical analysis of the biodiesel yields and the effect of selected parameters were discussed in the next session

3.7.2 Graphical analysis using design expert for statistical analysis

Design expert software of version11.0.0 was utilized for statistical analysis of for the experiment carried out in the laboratory using Box Behnken Design (BBD) for each parameters and the reply of measurement of the yields of biodiesel as discussed in section 3.7. For analysis of the experiment the inputs were feed in to the software and graphical analysis were shown properly for each experimental runs as recorded in Table3.6 below

3.8 ANOVA Analysis

Anova analysis is the very important parameter which indicates the factors that are significant and none significant for determination of the validity of the model showing experimental results accuracy. The significance of model is adequate as the value of P less than0.05% that indicates model accuracy and model terms significance. The higher the P values the less significance and accuracy of model terms. When lack of fit is none significant and high it demonstrates the model is fit as indicated in (Table 7).

Table 7 Anova analysis result

Source	Squares sum	df	Square mean	Value of F	of P Value	
Model	970.14	3	323	13	0.0003	significant
A-methanol to oil ratio	543	1	543	22	0.0004	
B-Temperature	259	1	259.32	10	0.0063	
C-catalyst loading	19.66	1	19.66	0.8005	0.3872	
Residual	319	13	24.56			
Lack of Fit	224	8	28.11	1.49	0.3434	not significant

Error	94	5	18.88
Cor Total	1289	16	

3.8.1 Comparison of actual and predicted biodiesel

The result from the experimental runs were analyzed based on actual value, predicted value, leverage and cook's distance ,the values from the software were tabulated in (Table 8).

The coefficient approximation represents the predict table alter in reaction per unit vary in issue charge when all outstanding factors are held unvarying. The interrupt in an orthogonal design is the in general normal response of all the runs. The differential factor (df) value which is unity indicates orthogonality of factors to each other and the value of VIF close to 1 shows the multi-colinearity of factors and the value less than ten (10) are also acceptable.

The equation in terms of real factors can be used to generate estimations about the response for given levels experimental runs. This equation should not be used to resolve the comparative impact of each factor because the coefficients are scaled to put up the units of each factor and the cut off is not at the center of the plan gap.

Table 8 Comparison of the result

Run Order	Actual Value	Predicted Value	Residual	Leverage	Cook's Distance
1	75.00	72.03	2.97	0.140	0.017
2	75.22	72.03	3.19	0.140	0.020
3	75.32	79.21	-3.89	0.061	0.011
4	85.00	87.39	-2.39	0.377	0.056
5	84.56	92.55	-7.99	0.268	0.326
6	71.32	77.19	-5.87	0.295	0.208
7	94.32	92.55	1.77	0.268	0.016
8	83.50	80.23	3.27	0.366	0.099
9	69.35	74.05	-4.70	0.361	0.198
10	73.32	72.03	1.29	0.140	0.003
11	60.00	65.87	-5.87	0.277	0.186
12	93.45	85.37	8.08	0.187	0.189
13	70.88	70.01	0.8653	0.333	0.006
14	83.50	79.21	4.29	0.061	0.013
15	85.00	86.39	-1.39	0.171	0.005
16	84.32	82.24	2.08	0.495[(1)]	0.085
17	83.50	79.21	4.29	0.061	0.013

3.8.2 Diagnostic Analysis of Model graphs of all factors

According to (Figure 7) demonstrates that as the biodiesel yield increases as methanol to oil ratio increases and illustrates the correlation between the three factors related to the biodiesel yield, as methanol to oil ratio increase biodiesel yield increases, as temperature increases biodiesel decrease and as catalyst saturated biodiesel nearly minimized.

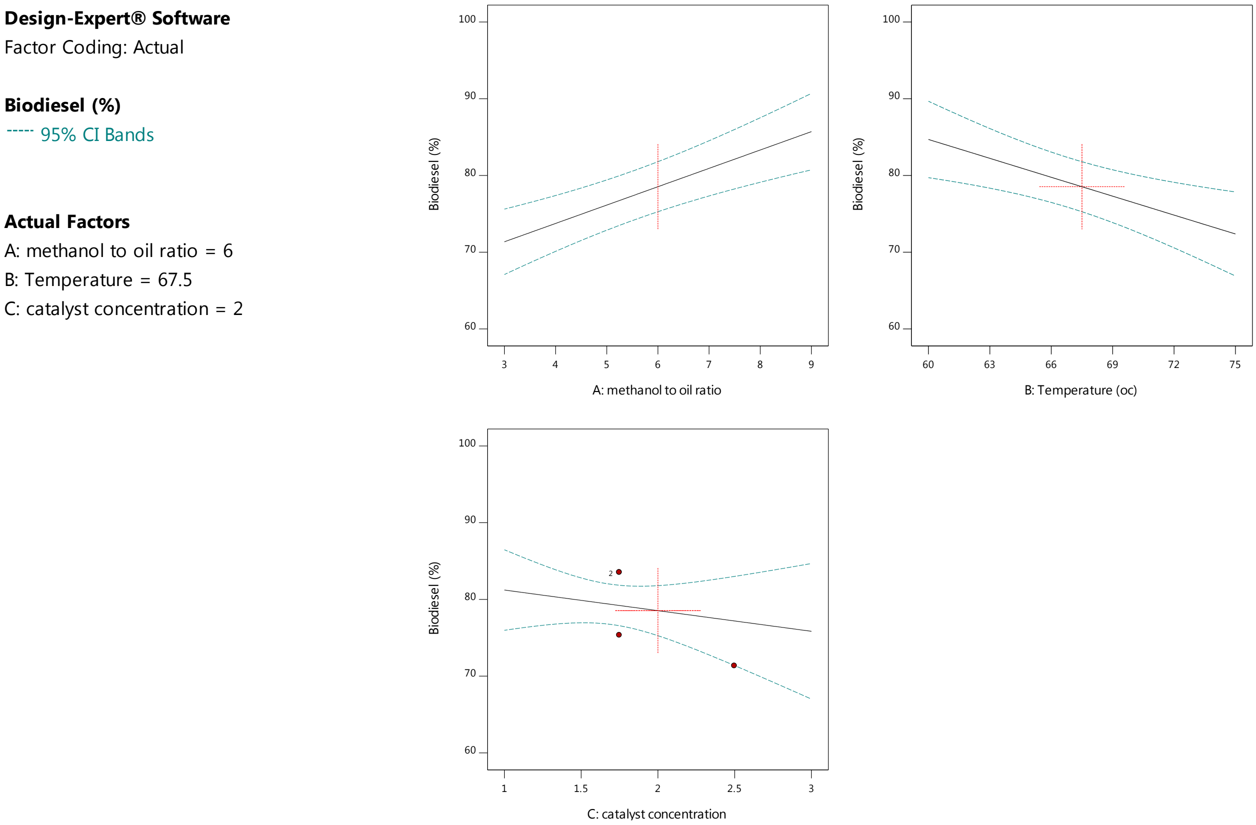

Figure 7: all factor graphs

3.8.3 Predicted versus actual graph Diagnosis model adequacy Test

After the development of models using the RSM, the significance tests using ANOVA were carried out in order to validate the model and its precision. The model was tested for adequacy by variance analysis

The low value of the coefficient of variation (CV=6.25%) indicates that the result of the integral model is reliable. The quality of the model fit was evaluated by the coefficient of determination (R^2), this value is calculated to be 0.7524 for the response. The value of R-square (R2) was 0.7524 this indicates that 75.24% of the total variation in biodiesel yield was attributed to the experimental variables studied. The closer the R2 value to unity, the better the model will be as it will give predicted values which are closer to the actual optimum values for the response.

From Table 4.8 the regression model was found to be highly significant with the correlation coefficients of determination of R-squared (R^2), adjusted R-squared and predicted R-squared having values of 0.7524, 0.6953 and 0.5637 respectively. The adjusted coefficient of determination (R2adj=0.6953) is also very high, supporting the significance of the model. However, quadratic terms of temperature, quadratic terms of catalyst concentration and cross product of methanol to oil ratio and temperature (AB) are found to be insignificant ($p>0.05$). Regression analysis of the experimental data also shows that methanol to oil ratio and catalyst concentration of reaction variables has positive and negative linear effects respectively on biodiesel yield.

Table 9: Model adequacy validation using Standard deviation, Mean and CV

Std. Dev.	4.96	R^2	0.7524
Mean	79.27	Adjusted R^2	0.6953
C.V. %	6.25	Predicted R^2	0.5637
		Adeq Precision	11.0981

3.8.4 Diagnostic Analysis Three D model graph of adequacy Test

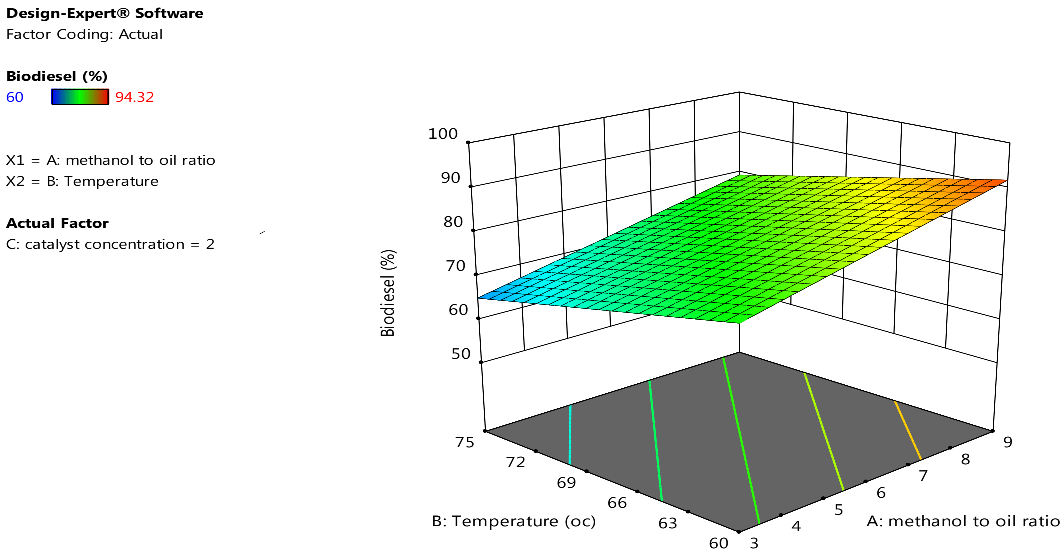

Figure 8: Three D Model graph and contours line graph

3.9 Discussion

In order to evaluate Soxhlet n-hexane as possible method for marula oil extraction, the impacts of temperature on the yield were evaluated and the oil was characterized. To determine the optimal safe operating conditions and study effects of temperature on the yield selected condition between 60 to 75^{o}c.Particle size effects on the yield were not studied. However, the material was ground prior to the extraction and after extraction the oil characteristics have been determined. The extraction period was dynamic and lasted from three hours to five hours based on temperature adjusted. Optimization of oil and Biodiesel were carried out using Design expert software and each characteristics of biodiesel were determined experimentally.

The effect of each process parameters was evaluated and physiochemical properties were determined. Catalyst loading beyond the optimum has the great effect on the production of biodiesel and it should maintain at the optimum. The biodiesel obtained in this study was 94.32% at the molar oil ratio of 9:1at the catalyst loading of 1.75%, this demonstrates that high biodiesel yield can be obtained at optimum catalyst loading.

3.10 Conclusion

Alternative Energy that is very essential to combat global warming particularly, the emission of Greenhouse gases is producing Biodiesel, which have less emission and have positive influence on environmental pollution. There are many resources useful for production of Biodiesel has future hope to solve dependence of fuels on fossil fuels.

From the experimental analysis carried out in this study reveals biodiesel produced from marula have acceptable quality. The marula seed oil was extracted using chemical extraction and chemically converted to biodiesel through transesterification to fatty acid methyl ether in the presence of KOH as catalyst. In this study different reaction parameter (reaction temperature, methanol to oil ratio and catalyst concentration) were analyzed to investigate biodiesel optimization. In the experimental analysis to optimize the effect of variable processes, for biodiesel production at optimum condition was evaluated using design expert software.

Generally, biodiesel is the most important alternative fuel to combat global warming manifesting itself throughout the world today and it is helpful for transportation to minimize carbon emission for vehicles.

Declaration

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Code of Availability: Not applicable

Conflict of Interest: The authors declared no conflict of interest

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Authors Contribution

BGE involves in research Design, Data collection, Data analysis and Draft manuscript

SKF works on literature and Data analysis. All authors read and approved their final manuscript

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