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Performance Evaluation of the Predictive Models for Free Fatty Acid Content and Biodiesel Yield from Non-Edible Oils

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***Abstract*— Non-edible oils pose a problem when used untreated for biodiesel production. This is due to the presence of high free fatty acid in the oils. Free Fatty Acid (FFA) content being a vital quality parameter for biodiesel production requires optimization to keep it at its minimum so as to maximize the yield of biodiesel. This paper on the one hand, reviews the current estimation methods for FFA content and biodiesel yield from non-edible oils. On the other hand, it evaluates the performance of the models developed for estimation of FFA content and biodiesel yield from non-edible oils. The study shows that, the statistical models developed for the prediction of biodiesel yield from the oils are reasonably good (judging by the high R^2 value, low maximum absolute error and worst case relative error < 6 %). It further shows that, though the models developed for estimation of FFA content of oils have good coefficient of determination values, p-values and F-values, on a relative error scale, the error (between the experimental and model prediction values) is significantly large (> 91%). It is concluded that, the models developed for estimation of biodiesel yield from oils with low FFA content are reliable. However, the current predictive models for FFA content lack the necessary accuracy when viewed on a relative error scale. It is therefore recommended that, for oils with low FFA content, the current optimization models be employed for prediction of biodiesel yield. However, other modelling techniques should be investigated for accurate and reliable estimation of FFA content of non-edible oils.**

Keywords*— Biodiesel production, non-edible oils, modelling, estimation, free fatty acid content, yield.

I. INTRODUCTION

Over the years, there has been much dependence on fossil fuel. This fuel, being a non-renewable energy resource, may soon become economically unavailable for domestic and industrial consumption. With the market price of petroleum becoming a concern, there is obviously a need for an alternative fuel source. Biodiesel (a high energy, organic compound that can be made from various vegetable oils) leads in the research for alternative sources of energy. Biodiesel is advantageous in the sense that, its feedstock is mostly renewable and its combustion significantly reduces carbon dioxide emission during its whole life cycle [1], [2]. However, the dependence on virgin oils as biodiesel feedstock raised sustainability concerns, such as the "Food vs. Fuel" debate [3]. In addition, it makes biodiesel less competitive in the fuel market due to the high cost of virgin oils [4]. Haas and Foglia [5] reported that the cost of the feedstock usually accounts for more than 80% of the total cost in biodiesel production.

Vegetable oils may be edible or non-edible. More than 95% of the renewable resources used for biodiesel production are edible oils [6] which in the long run may have serious effect on food availability and the cost of biodiesel, as it may be more expensive than petroleum diesel. In order to address these concerns, researches are currently focused on the use of non-edible oil plants in nature [7]; some of which are neem (*A. indica*), jathropha tree (*J.curcus*), Karanja (*P. pinnata*), etc. [8].

FFA content is reported [9] as one of the major parameters affecting biodiesel production from transesterification of non-edible oils. Traditionally, optimization models are developed to predict the FFA value of the oils. Thereafter, process conditions are established for minimizing the FFA value of the oil sample. Therefore, biodiesel is produced from the oil sample with minimum allowable FFA content.

This paper has two sided importance. On the one hand, it reviews the current estimation methods for FFA content and biodiesel yield from non-edible oils. On the other hand, it uses some statistical criteria to examine the performance of the models developed for estimation of FFA content and biodiesel yield. Therefore, the findings are expected to offer improved techniques for estimation of FFA content and biodiesel yield from non-edible oils.

II. LITERATURE REVIEW

A. Biodiesel Production

Biodiesel is formed when triglycerides (oils) react with alcohol in the presence of a catalyst, through a process called transesterification reaction. The catalyst can be an acid, alkaline or enzyme. For transesterification process, the alcohol commonly used is methanol. Methanol is less expensive than ethanol and it is easier to recover the unreacted methanol [10]. For alkaline catalyst, potassium hydroxide (KOH) or sodium hydroxide (NaOH) are often used because, they are cheaper and easier to handle in storage and transportation [10].

The major challenge associated with biodiesel production from non-edible feed stocks is the presence of high free fatty acid (FFA) content. Zhang and Co-workers [11] reported that, the triglycerides used in alkaline

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transesterification reactions should contain no more than 1% FFA; otherwise, saponification reaction will set in. This reaction prevents the separation of the ester formed from glycerin (a byproduct) which decreases the yield and requires refinement to remove soap [11], [12]. To overcome this problem, previous investigators [13] and [14] proposed a two-step acid–base catalyzed process. In the first step, the FFA in the oils was esterified with methanol using an acid catalyst while in the second step, the pre-treated product from the first step was transesterified using a base catalyst. Although the esterification reaction in the pre-treatment step changed the FFAs in oils into corresponding Fatty Acid Methyl Ester (FAME) otherwise known as biodiesel, unconverted FFA and triglycerides still remained in the pre-treated oil. Transesterification reaction was then performed to complete the reaction.

B. Model Formulation

Transesterification process leads to soap formation if the FFA content of the oil is not reduced to an acceptable value. To determine if the FFA value of the oil is within permissible limits, optimization models are usually developed as a function of process parameters. The process parameters may include alcohol-to-oil molar ratio, reaction temperature, catalyst concentration, reaction time and speed of agitation. Various experiments have been conducted that propose the optimum values for the process parameters mentioned above.

The main factors that drive the biodiesel conversion from non-edible oil (triglycerides) are usually studied using statistical methods. The aim is to determine the optimum parameters for the production of biodiesel. Statistical methods such as factorial design and response surface designs have been the traditional methods as they appear to offer the best parameter combination and greatly reduce the time spent on experiments.

C. Free Fatty Acid Estimation

As was mentioned in section I, the major parameters affecting biodiesel production by transesterification of vegetable oils with alcohol using base catalysts are the FFA and moisture content [9]. To minimize the FFA value of the oils, some mathematical (optimization) models have been developed to predict the FFA value of the oil at different conditions of the manipulated variables. For instance, in [15], [16], [17], second order models were developed for Jatropha Curcas Oil (JCO) as functions of four input parameters — Catalyst (H_2SO_4) concentration (%), Temperature, Time, and Methanol/Oil molar ratio. Also, Jahirul and Co-workers [18] demonstrated that a linear model appeared statistically more appropriate than the full quadratic model for describing the FFA content of Beauty Leaf (*Calophylum inophylum*) oil, as a function of three variables — methanol to oil molar ratio, M; catalyst concentration, C; and reaction temperature, T. Furthermore, Chai and Co-workers [4] developed a quadratic model that fitted experimental data better than the linear model for waste/used cooking oil with less than 15% FFA value. The statistical significance of these models in terms of the probability (p-) values, coefficient of determination (R^2) and the F-statistics were reported to be within acceptable ranges (p-value < 0.0001, R^2> 0.80, and F-value > 5) [16], [17].

C. Biodiesel Yield

The acid-pretreated oil (from the pretreatment esterification stage) at its optimum conditions is converted by means of an alkali catalyst (in the presence of an alcohol) to biodiesel. The goal of the transesterification stage is to maximize the yield of the biodiesel. Mathematical (optimization) models have been developed to predict the (maximum) yield of biodiesel from the pretreated oil sample at varying conditions of the manipulated variables. For instance, in [16], [17] response surface quadratic-based models with central composite rotatable design (CCRD) and central composite design (CCD), respectively, were developed to study the effects of these variables during alkali-catalyzed transesterification of the pretreated Jatropha oil. Also, Wang and Co-workers [14] employed a central composite design with response surface methodology to develop a second order model for biodiesel yield from trapped grease with 50% free fatty acid. Furthermore, Vicente and Co-workers [19] developed a second order model for the synthesis of methyl ester from sun flower oil. Moreover, in optimizing the transesterification reaction of canola oil and methanol (to minimize soap formation and maximize biodiesel yield), Singh and Co-workers [20] developed a second order model to predict the yield of biodiesel. The goodness of fit of these models in terms of the probability (p-) values, coefficient of determination (R^2) and the F-statistics were reported to be within acceptable ranges (p-value < 0.0001, R^2> 0.80, and F-value > 5)[16], [17] [21].

III. METHODOLOGY

Most of the models developed for estimation of FFA content and biodiesel yield have high R^2 values. It is known that sometimes, a model with high R^2 is not necessarily a very good model [22]. Therefore, in this study, in addition to coefficient of determination (R^2), performances of two selected models (case-studies: Betiku and Co-Workers [16], Goyal and Co-workers [17]) developed for estimating FFA content and biodiesel yield were determined by evaluating the absolute error, e_a and percent relative error, e_r . The values of these statistical criteria were calculated in Microsoft excel using the formulae (1) - (3).

$$e_a = |\text{Actual} - \text{Predicted}| \quad (1)$$

$$e_r = \frac{|\text{Actual}-\text{Predicted}|}{\text{Actual}} \times 100 \quad (2)$$

$$R^2 = 1 - \frac{\sum_{i=1}^{n}(y-y_p)^2}{\sum_{i=1}^{n}(y-\bar{y})^2} \quad (3)$$

where y_p is the predicted value of the independent variable and y is the experimental value. The experimental values were taken from [16] and [17].

IV. RESULTS AND DISCUSSION

The analyses of the experimental design for the development of the FFA content estimation model are presented in tables I and II. Table III summarizes the result of the analyses.

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TABLE I. CCRD ARRANGEMENT AND RESPONSES FOR ESTERIFICATION OF JATROPHA OIL [16]

Order	X_1	X_2	X_3	X_4	Exp.	Pred.	Rel. (%)	Abs. Error
1	-1	-1	-1	-1	3.27	2.73	16.51	0.54
2	-1	-1	1	1	2.14	1.65	22.90	0.49
3	0	0	0	0	0.99	1.35	36.36	0.36
4	1	-1	1	-1	2.26	0.97	57.08	1.29
5	0	0	0	2	1.97	2.39	21.32	0.42
6	1	-1	1	1	0.96	1.79	86.46	0.83
7	0	-2	0	0	5.02	0.76	84.86	4.26
8	-1	1	1	-1	1.07	0.86	19.63	0.21
9	1	1	1	1	0.56	2.88	414.26	2.32
10	0	0	2	0	0.85	1.8	111.76	0.95
11	-1	-1	-1	1	2.14	1.27	40.65	0.87
12	-2	0	0	0	1.24	0.89	28.23	0.35
13	0	0	0	0	1.3	2.37	82.31	1.07
14	0	2	0	0	0.79	1.77	124.05	0.98
15	0	0	0	0	1.18	0.51	56.78	0.67
16	-1	1	-1	-1	1.35	0.61	54.81	0.74
17	-1	1	-1	1	1.3	1.35	3.85	0.05
18	2	0	0	0	0.79	0.37	53.16	0.42
19	0	0	0	0	1.41	4.02	185.11	2.61
20	-1	1	1	1	0.96	1.48	54.17	0.52
21	0	0	0	-2	1.18	1.42	20.34	0.24
22	-1	-1	1	-1	1.92	0.81	57.81	1.11
23	0	0	0	0	1.02	1.47	44.12	0.45
24	0	0	0	0	1.41	1.38	2.13	0.03
25	1	1	-1	-1	1.69	1.22	27.81	0.47
26	1	1	1	-1	0.79	1.22	54.43	0.43
27	0	0	-2	0	1.69	1.22	27.81	0.47
28	1	-1	-1	1	1.97	1.22	38.07	0.75
29	1	1	-1	1	0.56	1.22	117.86	0.66
30	1	-1	-1	-1	0.4	1.22	205	0.82

TABLE II. CCD ARRANGEMENT AND RESPONSES FOR ESTERIFICATION OF JCO [17]

Run	A	B	C	D	Exp.	Pred.	Rel. (%)	Abs. Error
1	1	50	105	12	2.22	3.03	36.49	0.81
2	1.5	45	67.5	5.25	2.82	3.35	18.79	0.53
3	1.5	45	67.5	5.25	2.96	3.35	13.18	0.39
4	1.5	45	142.5	5.25	2.48	2.8	12.90	0.32
5	0.5	55	142.5	9.75	1.11	1.11	0	0
6	1	50	105	7.5	1.02	1.08	5.88	0.06
7	0.5	45	67.5	5.25	2.73	3.54	29.67	0.81
8	1.5	55	67.5	9.75	2.94	3.18	8.16	0.24
9	1	50	105	3	1.99	0.93	53.27	1.06
10	1.5	45	142.5	5.25	2.51	2.8	11.55	0.29
11	1.5	45	142.5	9.75	2.53	2.74	8.300	0.21
12	0.5	45	67.5	9.75	7.32	7.35	0.41	0.03
13	1	50	105	7.5	0.99	1.08	9.09	0.09
14	0.5	40	142.5	9.75	3.87	3.68	4.91	0.19
15	1	55	105	12	2.26	3.03	34.07	0.77
16	1	55	105	7.5	7.64	7.13	6.68	0.51
17	0.5	60	142.5	9.75	1.09	1.11	1.83	0.02
18	0.5	45	142.5	5.25	1.87	1.95	4.28	0.08
19	1	55	105	7.5	2.98	3.12	4.70	0.14
20	1.5	50	67.5	9.75	6.17	6.28	1.78	0.11
21	1.5	50	142.5	9.75	0.65	0.06	90.77	0.59
22	1	40	105	7.5	1.06	1.08	1.89	0.02
23	1	50	30	7.5	6.84	6.18	9.65	0.66
24	1	55	105	7.5	7.56	7.13	5.69	0.43
25	0	50	105	7.5	1.57	1.52	3.18	0.05
26	0.5	45	67.5	9.75	4.49	4.36	2.90	0.13
27	1	50	30	7.5	6.78	6.18	8.85	0.6
28	1.5	45	142.5	9.75	2.55	2.74	7.45	0.19
29	2	50	105	7.5	0.51	0.28	45.10	0.23
30	0.5	45	142.5	5.25	2.61	2.87	9.96	0.26
31	1	50	105	7.5	1.01	1.08	6.93	0.07
32	0.5	55	67.5	9.75	4.52	4.36	3.54	0.16
33	0.5	45	142.5	9.75	3.81	3.68	3.41	0.13
34	0.5	55	142.5	5.25	1.85	1.95	5.41	0.1
35	2	50	105	7.5	0.53	0.28	47.17	0.25
36	1	50	180	7.5	2.04	2.39	17.16	0.35
37	1.5	55	142.5	9.75	0.67	0.06	91.04	0.61
38	0.5	45	142.5	5.25	2.65	2.87	8.30	0.22
39	1	50	105	7.5	1.05	1.08	2.86	0.03
40	1	50	105	7.5	1.04	1.08	3.85	0.04
41	0.5	55	67.5	5.25	1.91	2.19	14.66	0.28
42	1.5	55	142.5	5.25	1.33	1.78	33.83	0.45
43	1	50	105	3	2.05	0.93	54.63	1.12
44	0.5	45	67.5	9.75	7.38	7.35	0.41	0.03
45	1.5	55	67.5	9.75	3.02	3.18	5.30	0.16
46	1	60	105	7.5	2.91	3.12	7.22	0.21
47	1.5	55	142.5	5.25	1.36	1.78	30.88	0.42
48	0.5	55	67.5	5.25	1.94	2.19	12.89	0.25
49	0.5	45	67.5	5.25	2.78	3.54	27.34	0.76
50	1.5	55	67.5	5.25	1.56	1.9	21.79	0.34
51	0	50	105	7.5	1.58	1.52	3.80	0.06
52	1	50	180	7.5	2.06	2.39	16.02	0.33
53	1.5	45	67.5	9.75	6.25	6.28	0.48	0.03
54	1.5	55	67.5	5.25	1.55	1.9	22.58	0.35

TABLE III. PERFORMANCE OF THE FFA CONTENT (ESTIMATION) MODELS

Case Study	Worst case, e_a	Worst case, e_r	R^2
[16]	4.26	414.286	0.001
[17]	1.12	91.045	0.958

For the first case (Betiku and Co-workers [16]), data for esterification of JCO (for prediction of FFA content of the oil) were analyzed for R^2, absolute and relative errors. Though the authors [16] did not state the value of R^2 for the model they developed for estimation of FFA content of JCO, the analysis shows that, the R^2 value is very low (0.001) and the model cannot account for the variation in the

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experimental data. The results of the analyses are as shown in table III. The relative error of over 400% for this model show further that this model can not accurately be used to estimate the FFA content of the JCO.

For the second case (Goyal and Co-workers [17]), data for developing model for estimation of FFA content were analyzed for R^2, absolute and relative errors. The value of R^2 calculated here, is in agreement with the R^2 reported in [17]. However, a model with high R^2 value (0.96), low maximum absolute error value, has maximum (worst case) relative error value of 91%. Therefore, the model with $R^2 = 0.96$, p-value < 0.0001 [17] and large worst case relative error cannot be said to be accurate/ reliable enough for estimation of FFA content of oil samples.

Tables IV and V give the analyses of the experimental design for the development of the biodiesel yield (prediction) model and table VI summaries the result of the analyses.

TABLE IV. CCRD AND EXPERIMENTAL, PREDICTED FOR FIVE-LEVEL-THREE-FACTOR RESPONSE SURFACE ANALYSIS FOR TRANSESTERIFICATION STEP [16].

order	X_1	X_2 (min)	X_3 (wt. %)	Exp.	Pred.	Rel. (%)	Abs. Error
1	-1	-1	-1	87.41	86.65	0.87	0.76
2	1	-1	-1	92.74	93.77	1.11	1.03
3	-1	1	-1	83.31	80.63	3.22	2.68
4	1	1	-1	87	87.75	0.86	0.75
5	-1	-1	1	91	88.52	2.73	2.48
6	1	-1	1	92.08	93.04	1.04	0.96
7	-1	1	1	87.81	85.06	3.13	2.75
8	1	1	1	90.54	89.58	1.06	0.96
9	-1.68	0	0	75.51	79.84	5.73	4.33
10	1.68	0	0	91.51	89.62	2.07	1.89
11	0	-1.68	0	91.43	91.35	0.087	0.08
12	0	1.68	0	80.85	83.38	3.13	2.53
13	0	0	-1.68	90	90.16	0.18	0.16
14	0	0	1.68	90.99	93.27	2.51	2.28
15	0	0	0	86.55	84.84	1.98	1.71
16	0	0	0	84.6	84.84	0.28	0.24
17	0	0	0	83.79	84.84	1.25	1.05
18	0	0	0	84	84.84	1	0.84
19	0	0	0	84.11	84.84	0.87	0.73
20	0	0	0	87.21	84.84	2.72	2.37
21	0	0	0	84.06	84.84	0.93	0.78

TABLE V. CCD ARRANGEMENT AND RESPONSES FOR TRANSESTERIFICATION OF JCO [17].

Run	A	B	C	D	Exp.	Pred.	Rel. (%)	Abs.
1	1	45	105	9	82.6	82.84	0.290	0.24
2	0	45	105	9	89.5	89.81	0.35	0.31
3	0.5	40	67.5	10.5	78.2	80.37	2.77	2.17
4	1.5	40	142.5	10.5	88.6	87.84	0.86	0.76
5	0.5	40	67.5	10.5	77.8	80.37	3.30	2.57
6	0.5	50	67.5	7.5	87.1	86.76	0.390	0.34
7	1.5	50	67.5	10.5	73.9	76.5	3.52	2.6
8	0.5	40	142.5	7.5	91	89.83	1.29	1.17
9	1	45	105	9	82.9	82.84	0.072	0.06
10	0.5	40	67.5	7.5	86.1	87.33	1.43	1.23
11	1	45	105	9	83.1	82.84	0.313	0.26
12	1	45	105	12	85.5	84.97	0.62	0.53
13	1	45	105	9	82.1	82.84	0.90	0.74
14	1	45	180	9	93.4	92.65	0.803	0.75
15	0.5	50	142.5	10.5	97.8	99.99	2.24	2.19
16	1	45	30	9	78.2	79.56	1.74	1.36
17	1.5	50	142.5	10.5	88.3	87.1	1.36	1.2
18	0.5	50	67.5	10.5	90	87.17	3.14	2.83
19	0.5	40	142.5	10.5	91.8	91.86	0.065	0.06
20	1.5	40	67.5	7.5	85.7	84.18	1.77	1.52
21	0.5	50	142.5	7.5	90	90.98	1.089	0.98
22	1	35	105	9	92.4	91	1.52	1.4
23	1.5	40	142.5	7.5	81.6	84.05	3.0025	2.45
24	1.5	40	142.5	10.5	83.8	87.84	4.82	4.04
25	1	45	105	12	85.9	84.97	1.08	0.93
26	0.5	50	67.5	10.5	90.4	87.17	3.57	3.23
27	1.5	40	67.5	10.5	80	78.97	1.29	1.03
28	2	45	105	9	73	73.37	0.51	0.37
29	1	55	105	9	88.2	89.69	1.69	1.49
30	1.5	50	142.5	7.5	77	75.94	1.377	1.06
31	1.5	40	67.5	7.5	85.2	84.18	1.20	1.02
32	1	45	105	9	82.4	82.84	0.534	0.44
33	1	55	105	9	87.9	89.69	2.04	1.79
34	1.5	50	67.5	10.5	74.1	76.5	3.24	2.4
35	1.5	50	67.5	7.5	74.8	74.34	0.61	0.46
36	1.5	50	142.5	7.5	77.3	75.94	1.76	1.36
37	1.5	40	67.5	10.5	80.2	78.97	1.53	1.23
38	1	45	105	9	82.8	82.84	0.048	0.04
39	0.5	50	67.5	7.5	87.5	86.76	0.846	0.74
40	2	45	105	9	73.2	73.37	0.23	0.17
41	1	45	105	6	79.5	80.76	1.58	1.26
42	1.5	50	67.5	7.5	74.5	74.34	0.215	0.16
43	0	45	105	9	89.9	89.81	0.100	0.09
44	1.5	40	142.5	7.5	81.2	84.05	3.51	2.85
45	0.5	50	142.5	7.5	90.4	90.98	0.641	0.58
46	0.5	50	142.5	7.5	98.2	99.98	1.81	1.78
47	1	35	105	10.5	92.1	91	1.19	1.1
48	1	45	105	9	79.8	80.76	1.20	0.96
49	0.5	40	67.5	6	86.5	87.33	0.96	0.83
50	0.5	40	142.5	7.5	92.1	91.86	0.261	0.24
51	1	45	30	10.5	78.5	79.56	1.350	1.06
52	0.5	40	142.5	7.5	91.4	89.83	1.71	1.57
53	1.5	50	142.5	10.5	88.7	87.1	1.80	1.6
54	1	45	180	9	93.7	92.65	1.12	1.05

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TABLE VI. PERFORMANCE OF THE BIODIESEL YIELD (ESTIMATION) MODELS

Case Study	*Worst case, ε_a*	*Worst case, ε_r*	*R^2*
[16]	*4.33*	*5.734*	*0.814*
[17]	*4.04*	*4.821*	*0.946*

For the first case (Betiku and Co-workers [16]), data for transesterification of pretreated JCO (for prediction of biodiesel yield from the oil) were analyzed for R^2, absolute and relative errors. The results of the analyses are as shown in table VI. Though the authors [16] reported R^2 value (0.9302) higher than what was calculated here (R^2 = 0.814), the worst case relative error is less than 6%. This means, the model can be used for prediction of biodiesel yield since it has R^2 value which is within acceptable range [16], [17], [21] and low worst case relative error. For the second case (Goyal and Co-workers [17), data used for developing their model for prediction of biodiesel yield from the pretreated oil were analyzed for R^2, absolute and relative errors and the results are presented in table VI. The value of R^2 calculated here, is in agreement with the R^2 reported [17]. Also, table VI shows that the worst case relative error is less than 5%. This indicates that the reported model can provide reliable estimation of biodiesel yield.

V. CONCLUSION

Non-edible oils pose a problem when used untreated for biodiesel production. This is as a result of the high free fatty acid content of the oils. This is why optimization techniques are used for minimizing the free fatty acid (FFA) content of the oil and for maximizing the yield of biodiesel from the oil. This study shows that, the statistical models developed for the prediction of biodiesel yield from the oils are reasonably good (judging by the high R^2 value, low maximum absolute error and worst case relative error < 6 %). It further shows that though the models developed for estimation of FFA content of oils have good coefficient of determination values, p-values and F-values, on a relative error scale, the error (between the experimental and model prediction values) is significantly large (> 91%). The study therefore concludes that, the models developed for estimation of biodiesel yield from oils with low FFA content are reliable. However, the current estimation method for FFA content lacks the necessary accuracy when viewed on a relative error scale. It is therefore recommended that for oils with low FFA content, the current optimization models be employed for prediction of biodiesel yield. However, other modelling techniques should be investigated for accurate and reliable estimation of FFA content of non-edible oils.

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