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Synthesis and RSM Optimization of Methyl Ester from Waste Fish Oil by Transesterification Using Potassium Hydroxide Catalyst

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Abstract

Renewable energy is vital for countering fossil fuel depletion and pollution, offering flexibility through its diverse and sustainable sources. Biofuels, derived from renewable organic materials, are a major renewable resource with a low carbon footprint, non-toxic, biodegradable, and sulfur-free, reducing emissions and health risks. Salmon oils pose environmental hazards and are challenging to dispose of properly. Therefore, it was proposed to convert them into biodiesel fuel as a sustainable solution. The process applied transesterification, employing methanol and potassium hydroxide as the catalyst to convert the oil into methyl ester. The impact of four parameters—reaction time, temperature, methanol and catalyst concentration were investigated. Experiments were conducted through various ranges for each variable: temperature was studied from 40 °C to 65 °C in increments of 5 °C, methanol concentrations from 10 % to 20 % in increments of 5 % wt./wt., catalyst concentration from 0.25 % to 1.5 % in increments of 0.25 % wt./wt., and reaction time from 15 to 90 minutes in increments of 15 minutes. The optimal conditions were determined using the Response Surface Methodology (RSM) model in Design Expert 13, based on optimization procedures for the settings of factorial variables, aiming for the minimum temperature, time, and catalyst concentration, with a methanol range of 10 % to 20 % by wt. The optimal production of fatty acid methyl esters (FAME) occurred at a reaction temperature of 50 °C, with 20% methanol by weight and 0.875% potassium hydroxide by weight, for 60 minutes, getting a maximum yield of 89.07%.

Keywords: Waste fish oil, biodiesel synthesis, optimization, homogeneous catalyst, TLC technique.

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1. Introduction

Fossil fuels, including coal, oil, and natural gas, have been the backbone of industrialization and economic development [1] [2][3]. However, they come with significant environmental and health costs such as Climate Change due to the emission of carbon dioxide (CO_2), air Pollution linked to millions of premature deaths annually and non-renewable fossil fuels which are finite resources considered unsustainable in the long term [4][5][2]. While fossil fuels have powered our economies for centuries, their negative impacts on the environment and health are driving the shift towards renewable energy sources like biodiesel, which offer a cleaner, sustainable alternative [6][7].

Biodiesel represents a renewable ecofriendly fuel that is integral to the amelioration of environmental detriments associated with petrochemical hydrocarbons [8][9]. Biodiesel is a renewable fuel made from organic materials, typically derived from vegetable oils, animal fats, or recycled cooking grease[10][11]. Biodiesel offers a promising renewable energy solution that can help reduce dependence on fossil fuels and mitigate environmental impacts associated with transportation [12] [13]. Utilizing waste fish oil for biodiesel not only provides a renewable energy source but also addresses environmental concerns associated with the disposal of fish processing waste [14][15]. The conversion of waste fish oil to biodiesel represents a promising avenue for sustainable energy production, offering a dual benefit of waste reduction and energy generation [16] [17].

Roughly 60,000 metric tons of salmon heads are produced annually as a byproduct of processing pink and red salmon in Alaska. A significant portion of the oil in some salmon is located in the head, which has a lipid content ranging from 11 to 18 percent. Salmon oil is a rich source of omega-3 polyunsaturated fatty acids, particularly eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) [18] [19]. In Egypt, according to the statements of the smoked salmon production and packaging factory in Badr City A significant quantity of oil is extracted from smoked salmon waste, with production averaging around 3 tons per month. This volume constitutes approximately 30-35% of the total waste generated from a single salmon fish. the processing establishments specializing in the canning and smoking of piscine products accrue considerable

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quantities of non-consumable lipids. These byproducts precipitate notable ecological deterioration and concomitant public health risks[20][21]. In response, initiatives have been instituted to repurpose these lipidic byproducts into renewable bioenergy vectors, specifically biodiesel, as a viable surrogate to conventional hydrocarbon-based energy sources. This paradigm not only mitigates waste management challenges but also augments the diversification of energy portfolios, thereby attenuating the ecological footprint of energy production [22] [23]. Waste fish oil is increasingly recognized as a valuable feedstock for biodiesel production due to its abundance and sustainability. Waste fish oil, a byproduct of the fish processing industry, is rich in triglycerides suitable for biodiesel production [24][25]. The decision to use salmon oil alone in this context is due to its unique composition and the presence of special compounds that distinguish it from vegetable oils and used cooking oils. Salmon oil is rich in omega-3 fatty acids, particularly eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA), These compounds are not as prevalent in vegetable oils, which typically have higher levels of omega-6 fatty acids [26]. Furthermore, the composition of salmon oil may offer advantages in terms of biodiesel production. The fatty acid profile of salmon oil could potentially lead to biodiesel with different physical and chemical properties compared to biodiesel derived from vegetable oils [27] [28].

Recent research has delved into biodiesel synthesis from fish waste oil, with Yahyaee et al. [29] investigation offering a comprehensive analysis of the current and potential roles of waste fish oil as a renewable energy source in Iran. This study focused on the transesterification reaction dynamics involving methanol, potassium hydroxide, and fish oil. The experimental findings revealed a biodiesel production yield of 0.9 liters per liter of fish oil, shedding light on the viability of this process. Furthermore, Girish et.al[30]., explored an environmentally friendly process for producing biodiesel from fish waste. The fat is extracted using water as a solvent, followed by saponification to remove free fatty acids. Transesterification is then performed using methanol and potassium hydroxide, resulting in a biodiesel yield of up to 85%. The biodiesel produced meets ASTM standards In the 2019 study by Anand Kumar et al. [31], the authors delineated the utilization of Indian oil sardine fish as an economical substrate for biodiesel generation via a KOH-catalyzed transesterification mechanism. The reaction was conducted at an elevated temperature of 150 °C, which expedited the process, curtailing the typical transesterification duration from 60–120 minutes to a mere 25 minutes. The research identified the optimal reaction milieu to achieve a biodiesel conversion efficiency of 96.57%, which included a methanol volume fraction of 20%, a KOH weight percentage of 1.25%, and a reaction time of 25 minutes.

Driven by the demand for sustainable alternatives and the awareness that much research concentrates on increasing biodiesel yield without addressing the significance of achieving optimal conversion efficiency, which affects biodiesel's performance and emissions in engines, this study seeks to enhance both the yield and conversion efficiency of biodiesel derived from oil produced during the salmon smoking process.

To achieve this, the research explores the optimization of biodiesel production from oil produced during the salmon smoking process, using a homogeneous alkali catalyst. The goal is to find the best conditions for converting this oil into biodiesel, considering the unique challenges posed by its composition. The study meticulously examined various parameters influencing biodiesel synthesis, including reaction temperature, duration, and the quantities of alkaline catalyst and methanol employed. Utilizing the Design Expert software, the research endeavored to ascertain the optimal conditions conducive to the most efficacious biodiesel production.

1 Material and Methods

1.1 Materials

The lipid extract obtained through the smoking and packaging of Salmon from company in Badr City, Egypt, underwent a physicochemical analysis to ascertain its fatty acid composition and oil properties. Potassium hydroxide ranging between 85–100% purity, and methanol with a purity of 99.5% were procured from Sigma Aldrich Co.

1.2 Biodiesel synthesis procedure

The experimental procedures were conducted in a 100 cm^3 glass reactor, positioned atop a magnetic stirrer equipped with a heating plate, and operated at ambient pressure. Initially, A sample of waste fish oil (WFO) was introduced into the reactor and heated to a predetermined temperature. Following this, a mixture of methanol and KOH catalyst—selected for its potent basicity and superior catalytic efficacy—was amalgamated with the preheated oil. The study meticulously explored a range of variables to ascertain the ideal conditions for the reaction, such as reaction temperature, duration, catalyst, and methanol concentration [32].

After post-reaction completed according to fig. (1), the products were settled for a period of 1 h to facilitate phase separation, after which it was decanted into a separation funnel to partition the biodiesel from the glycerol. The biodiesel layer underwent several washes with hot water. After the washing, the biodiesel was desiccated on a heating plate set at 110 °C to eliminate any residual moisture. The biodiesel yield was quantified employing Equation (1) [33] [34].

$$\text{Yield\%} = \frac{\text{mass of biodiesel,gm}}{\text{mass of oil,gm}} \qquad \text{Eq. (1)}$$

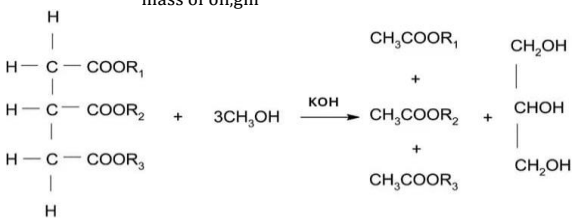

Figure 1: Chemical reaction for transesterification reaction

1.3 Characterization of the optimum WFO biodiesel sample

The optimal sample of WFO biodiesel underwent physicochemical property assessments, including tests for kinematic viscosity, density, flash point, acid value, and pour point. These tests were conducted using potassium hydroxide (KOH) as a base homogeneous catalyst, chosen through design expert software, following ASTM standards.

1.4 Modeling and optimization

In this study, the Box-Behnken design from Response Surface Methodology (RSM), as implemented in Design Expert 13, was employed. This approach facilitated the modeling and analysis of how four pivotal factors such as temperature, time, wt.% KOH catalyst, and wt.% methanol concentration—influence the yield of biodiesel [35][36]. An extensive Analysis of Variance (ANOVA) was conducted to assess the significance of each factor and their interaction effects on the yield. Moreover, numerical optimization within Design Expert was utilized to determine the optimal conditions that maximize biodiesel yield. Table 1 presents 51 experimental runs, with factor values ranging within specified lower and upper limits.

Table 1: The Experimental runs design

Run	F1: Temperature, ^{o}C	F2: Time, min.	F3: Wt.% KOH	F4: Wt.% methanol	Response Yield, %
1	50	60	1	10	86.219
2	70	60	1	10	82.539
3	90	60	1	10	77.739
4	110	60	1	10	71.819
5	130	60	1	10	64.779
6	50	60	1	15	87
7	70	60	1	15	83.728
8	90	60	1	15	79.248
9	110	60	1	15	73.248
10	130	60	1	15	65.728
11	50	60	1	20	88
12	70	60	1	20	85.836
13	90	60	1	20	82.276
14	110	60	1	20	76.476
15	130	60	1	20	68.436
16	50	60	0.25	10	12.9996
17	50	60	0.5	10	58.7215
18	50	60	0.75	10	81.5546
19	50	60	1	10	81.499
20	50	60	1.25	10	58.5546
21	50	60	1.5	10	12.7215
22	50	60	0.25	15	15.5005
23	50	60	0.5	15	60.778
24	50	60	0.75	15	83.6105
25	50	60	1	15	83.998
26	50	60	1.25	15	61.9405
27	50	60	1.5	15	17.438
28	50	60	0.25	20	16.5006
29	50	60	0.5	20	62.6675
30	50	60	0.75	20	85.5006
31	50	60	1	20	85
32	50	60	1.25	20	61.1656
33	50	60	1.5	20	13.9975
34	50	15	0.875	10	53.3555
35	50	30	0.875	10	67.496
36	50	45	0.875	10	76.3715
37	50	60	0.875	10	79.982
38	50	75	0.875	10	78.3275
39	50	90	0.875	10	71.408
40	50	15	0.875	15	63.75
41	50	30	0.875	15	75
42	50	45	0.875	15	81.75
43	50	60	0.875	15	84
44	50	75	0.875	15	81.75
45	50	90	0.875	15	75
46	50	15	0.875	20	70.7475
47	50	30	0.875	20	80.49
48	50	45	0.875	20	86.2275
49	50	60	0.875	20	87.96
50	50	75	0.875	20	85.6875
51	50	90	0.875	20	79.41

2 Result and discussion

2.1 Effect of reaction temperature on biodiesel produced.

Figure 2 examined the impact of diverse temperatures (ranging from 40 to 65 C in 5-degree increments) alongside different methanol concentrations (10%, 15%, 20%) during transesterification with a 1 wt.% KOH catalyst over a duration of 1 hour. At the beginning, increasing temperature increased biodiesel production. This enhanced solubility allows for better mixing and increased contact between the methanol and oil molecules, leading to more efficient transesterification reactions. Subsequently, a significant reduction was noted due to the evaporation of methanol at elevated temperatures. As methanol concentrations increase, the supply of reactants augments, enabling a higher conversion rate of triglycerides into biodiesel. However, this conversion rate plummeted with rising temperature due to the vaporization of alcohol at high temperatures [33] [35]. Results showed that the optimum biodiesel yield was at 50ºC at 20% wt. methanol.

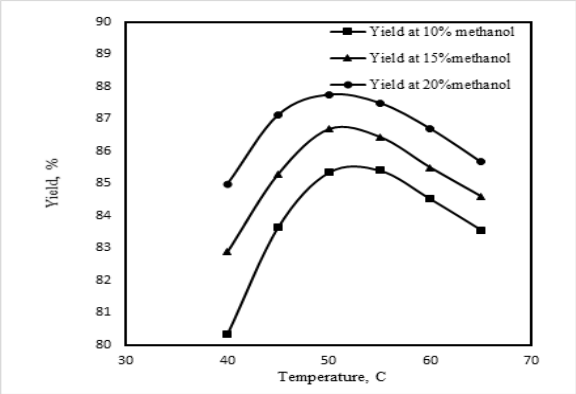

Figure 2: Effect of reaction temperature on biodiesel produced.

2.2 Effect of catalyst concentration on biodiesel produced.

Figure 3 depicted the influence of KOH catalyst levels (ranging from 0.25 to 1.5 wt.% in increments of 0.25) in conjunction with different methanol concentrations (10%, 15%, 20%) during transesterification at an optimal temperature of 50°C for the duration of 1 hour. Initially the biodiesel yield was decrease at low catalyst concentration at 20% methanol concentration because at lower catalyst concentrations, the reaction may proceed at a slower rate, leading to incomplete conversion of triglycerides to biodiesel and glycerol. As the catalyst concentration increases to 0.85% wt., the catalytic activity becomes adequate to expedite the reaction rate, improving the conversion of triglycerides into biodiesel. However, beyond this point, with further increases in KOH concentration, saponification reactions begin to dominate over the intended biodiesel production reaction, leading to a decrease in biodiesel yield [37]. Consequently, the findings indicated that the best yield occurred at a concentration of 0.85% weight with 20% weight methanol.

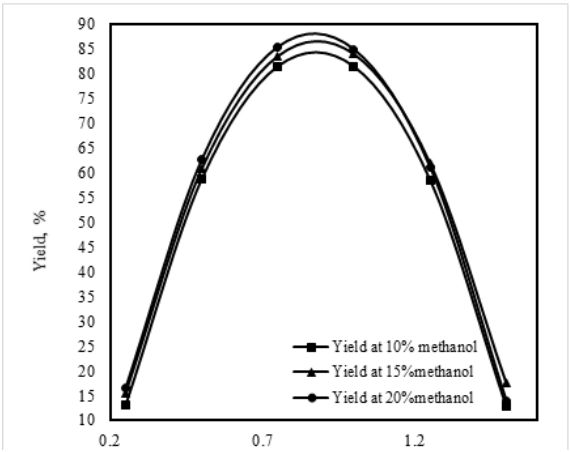

Figure 3: Effect of catalyst concentration on biodiesel produced

2.3 Effect of reaction time on biodiesel produced

In Figure 4, the influence of reaction duration on biodiesel mass yield was examined. Various time intervals (ranging from 15 to 90 minutes in 15-minute increments) were explored alongside different methanol concentrations (10%, 15%, 20%) during transesterification at the ideal temperature of 50°C and with 0.85% weight of KOH. It was observed that increasing time with increasing methanol concentration increase the biodiesel yield as the reaction time and methanol concentration rise more triglycerides are converted to biodiesel due to prolonged exposure to the catalyst and reactants. This extended duration allows for greater interaction between methanol and the oil, leading to increased biodiesel production. After beyond 60 minutes, the negative impact of side reactions outweighs the benefits of extended reaction times and higher methanol concentrations, resulting in a decline in biodiesel yield. The decline in FAME content is associated with alcohol evaporation and the reversible characteristics of the transesterification reaction. Additionally, high methanol concentrations can trigger saponification reactions, resulting in the formation of soap, which diverts the reaction pathway away from biodiesel production, thereby diminishing the total biodiesel yield [38]. Consequently, the findings indicated that the best yield occurred at a reaction time of 60 min with 20% weight methanol.

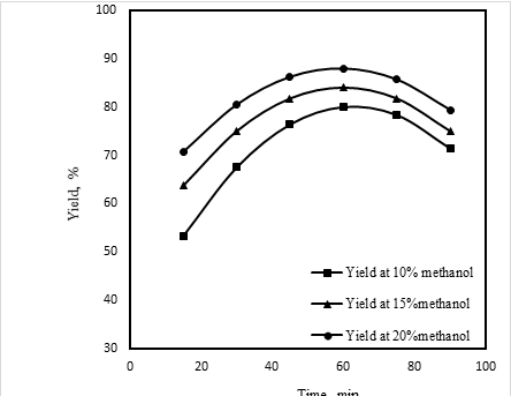

Figure 4: Effect of reaction time on biodiesel produced.

2.4 Optimization and modeling for the biodiesel yield

2.4.1 Modeling using ANOVA analysis

The yield model underwent evaluation through a series of experimental trials. Design Expert 13 software crafted models to probe the interplay between process variables and essential outcomes. ANOVA was executed to gauge the models' robustness and relevance, with a particular emphasis on F-values. The quadratic model emerged as the most fitting representation for the yield model. Nonetheless, certain model components were found to be statistically non-significant (with p-values exceeding 0.01) [39]. To refine the models, these non-significant elements were excised, yielding a more streamlined and precise depiction of the biodiesel production process. The refined model for biodiesel yield is encapsulated in Equation (2), as delineated by the Design Expert software. The ANOVA findings are encapsulated in Tables 2.

Table 2: ANOVA Analysis results for the response biodiesel yield

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	25849.15	7	3692.74	707.22	< 0.0001	significant
A-Temperature	13.18	1	13.18	2.52	0.1190	
B-KOH catalyst	0.3687	1	0.3687	0.0706	0.05916	
C-Methanol	314.97	1	314.97	60.32	< 0.0001	
D-Time	402.62	1	402.62	77.11	< 0.0001	
CD	63.15	1	63.15	12.09	0.0011	
B^2	24793.30	1	24793.30	4748.36	< 0.0001	
D^2	1392.50	1	1392.50	266.69	< 0.0001	
Residual	240.19	46	5.22			
R^2	0.992					

Yield, % = --126.37688+ 0.09823* Temperature +320.6216* wt.% KOH + +1.54530* wt.% Methanol+ 1.746985* Time - 0.017426*Methanol wt.%* Time - 183.02443*KOH catalyst² -0.012415*Time² Eq. (2)

Statistical evaluations validated the selected model's capability to forecast enhancements in biodiesel yield across the examined variable spectrum. The model's robustness is underscored by a high coefficient of determination (R^2), recorded at 0.99. Such a figure reflects a substantial alignment between the model's predictions and the empirical data, ensuring dependable forecasts within the scope of the variables assessed.

***2.4.2* Effect of two various factors on the biodiesel yield**

Figure 5a illustrates the correlation between the reaction temperature and the duration on biodiesel production, depicted on a three-dimensional graph. The x-axis indicates the temperature (A), while the z-axis shows the reaction time (D). The graph reveals a direct correlation; as both the temperature and the reaction time increase, the yield of biodiesel also rises then slightly decrease [40].

Figure 5b demonstrates the connection between the reaction temperature and the KOH concentration on biodiesel production, also on a three-dimensional graph. The x-axis denotes the temperature (A), and the z-axis represents the KOH concentration (B). Contrary to Figure 5a, this graph indicates an inverse relationship; an increase in temperature coupled with a rise in KOH concentration results in increase biodiesel yield then a sharp decrease was observed [40].

Figure 5C shows the impact of reaction temperature(A) and methanol concentration (C) on biodiesel yield, again using a three-dimensional curve. Here, a direct relationship is observed; higher temperatures and greater methanol concentrations lead to an enhanced biodiesel yield.

Figure 5d outlines how the methanol concentration (C) and Time (D) affect biodiesel production. This figure suggests a direct relationship between reaction time and methanol concentration; as both increases, so does the biodiesel yield[40] [41].

Figure 5e presents the interplay between reaction time and KOH concentration (weight percentage) on biodiesel yield, visualized on a three-dimensional curve. The x-axis is for time (A), and the z-axis for KOH concentration (B). The figure notes an inverse relationship; a higher reaction time along with an increased KOH concentration leads to a higher biodiesel yield up to 1% then sharp decrease was observed with increasing catalyst concentration [40] [41].

Lastly, figure 5f depicts the relationship between methanol concentration and KOH concentration on biodiesel yield. It is observed that there is a direct relationship between the KOH and methanol concentrations on the biodiesel yield; an increase in both initially boosts the yield, followed by sharp decrease with increasing catalyst concentration [42].

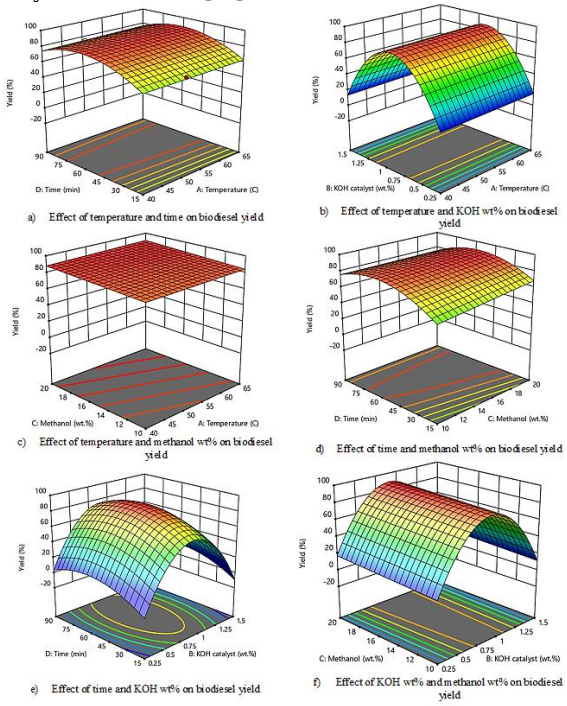

Figure 5: 3D response surface plot illustrating interaction effects on the biodiesel yield.

2.4.3 Determination of the best criteria using Design Expert software

Response Surface Methodology (RSM) is a collection of mathematical and statistical techniques useful for developing, improving, and optimizing processes [43]. In this paper, Design Expert software was used to determine the optimum conditions for a process.

The appropriate criteria were carefully chosen for this process to ensure the highest biodiesel yield at the minimal temperature, which is recommended due to the high energy consumption and consequent financial implications that necessitate its limited use. The other factorials were time ranging from 15 to 90 min, and catalyst concentration ranging from 0.25 to 2%, with a specific focus on methanol concentration ranging from 10 to 20% by weight. For a maximum biodiesel yield and compared to the experimental calculation as present in Table 3.

It was found that the maximum biodiesel yield was 89.07%. These values were achieved at a temperature of 40 °C, a reaction time of 60 min, and a catalyst concentration of 0.88 wt.% KOH and 20 wt.% methanol as shown in Figure 6. The red dots in the figure indicate the optimal input factors values, while the blue color signifies the maximum outcome value**.** By comparing the optimal values obtained from Design Expert with the experimental values, it was found that they agree with each other with an error rate of less than 1.2%.

Table 3: The optimization for various factors on the maximum biodiesel yield

Type	Temperature °C	Time min.	Wt.% KOH	Wt.% methanol	Yield %	Desirability %	Error%
Experimental	50	60	0.875	20	87.96	--	1.2%
DX numerical optimization	50	60	0.875	20	89.07	100	

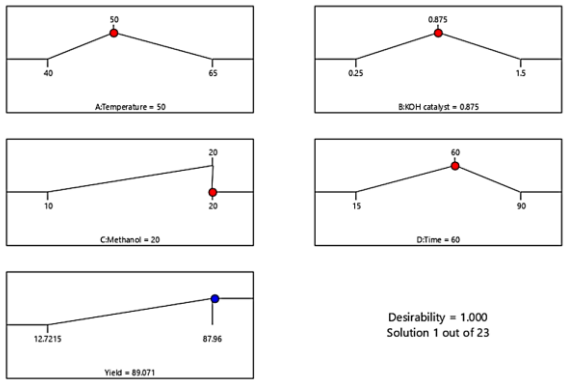

Figure 6: Ramps graph for maximum biodiesel yield for optimization DX @ software

2.5 Examination of the optimum biodiesel produced

The optimum biodiesel sample was fabricated utilizing ideal synthesis parameters, maintaining a reaction milieu at 50 °C, employing 20% by weight of methanol, and incorporating 0.85% by weight of potassium hydroxide as a catalytic agent over a period of 60 minutes. After synthesis, the biodiesel was subjected to an exhaustive suite of analytical procedures, the details of which are systematically enumerated in Table 4. Density, kinematic viscosity, acid value, pour point, and flash point, were measured in accordance with the appropriate ASTM Standards biodiesel D6751 and compared with raw WFO and commercial diesel fuel [35]. It was observed that the biodiesel sample produced from fish oil waste (B100) meets the specifications of the ASTM D6751 standard for biodiesel. Its properties, such as density, viscosity, and acid value, are comparable to those of commercial diesel (D100). Notably, the pour point and flash point of the B100 sample are superior to those of the D100 sample and fall within the acceptable ranges defined by the ASTM standard for biodiesel. The results obtained indicate a favorable alignment with the standards for engine combustion, implying that this biodiesel could fulfill the criteria necessary for effective engine functioning.

Table 4: Analytical Contrast of raw WFO, Synthesized Biodiesel (B100), commercial Diesel (D100), and ASTM D6751 Standardized Biodiesel

Parameter	Raw WFO	Commercial diesel fuel (D100) [44]	Pure optimum biodiesel sample produce (B100)	ASTM Standards biodiesel D6751 [35]
Density at 15 °C, g/ml	0.92	0.82	0.83	0.86 - 0.9
Viscosity at 40 °C ,(mm^2 /s)	30.5	1.3-2.4	9.5	1.9 - 6
Acid value, mg KOH/g biodiesel	5.6	0.07	0.09	Max. 0.5
Pour point, °C	11	6	-1	-5 - 10
Flash point, °C	145	90	130	Min. 100
Water content % wt.	0.03	0.05	0.01	Max. 0.05

3 Conclusion

This investigation sought to mitigate the escalating fuel demand and to identify alternative energy sources that could promote sustainable development. The objective was to repurpose waste fish oils into biodiesel fuel, due to the substantial environmental hazards associated with their inappropriate disposal and unsafe handling. The transesterification process employed potassium hydroxide and methanol to convert the waste fish oil into biodiesel. Key variables such as temperature, methanol concentration, and catalyst loading were examined. The Response Surface Methodology (RSM) was utilized to analyze the factors influencing biodiesel yield and its conversion at the minimum temperature and reaction time, which are the most economically impactful parameters. The optimum conditions for synthesizing fatty acid methyl esters (FAME) were established at a reaction temperature of 50 °C, with 20 wt.% methanol and 0.875 wt.% KOH, over a reaction time of 60 minutes, yielding a maximal biodiesel yield of 89.07 %. Upon comparing the optimal conditions Experimentally with the best outcome predicted by the RSM program for the selected criteria, it was observed that

the optimum conditions were nearly identical, resulting in the same amount of biodiesel production, with a error difference of 1.2%. The fuel obtained under these optimal conditions was subjected to chemical and physical analyses, which corroborated its quality in accordance with established ASTM Standards biodiesel D6751 and commercial diesel.

Recommendation and future work

It is very important to enhance biodiesel production through a multi-stage conversion process, which includes pre-treating oil to remove impurities or start the conversion of triglycerides into biodiesel, followed by a second stage to complete the process. This method is beneficial for oils with high FFA content, preventing soap formation and improving biodiesel separation. Additionally, the research also investigates the use of nano catalysts, which offer a high surface area-to-volume ratio and could increase catalytic activity, leading to more efficient biodiesel production with potential cost reductions and sustainability improvements.

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