Original Paper

Fishing industries' oily wastewater biodiesel performance

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Received: 14 March 2023 | **Accepted:** 13 May 2023 | **Published:** 4 June 2023

Keywords: biodiesel, performance, quality, sustainability

Abstract. *In the context of developing biodiesel as an alternative energy source for replacing fossil diesel, this study aimed to assess how physicochemical factors affected the efficiency of the process for producing biodiesel from oily residues in fishing industry effluent. The oil extracted from the grease traps was initially characterized for the stabilization of the process's influential parameters, obtaining viscosities of 38 mPa/s, densities of 0.93*

g/ml, and saponification indices of 260.40 mg KOH/g. It was then necessary to neutralize the oil and dry it to reduce the oil's acidity to 0.97% and its humidity to 0.03%, in order to meet the conditions for subsequent transesterification. This led to the conclusion that T7 (80 ºC, 9:1, and 0.8%) was the best treatment. It produced a 94% performance of biodiesel extraction and had the following properties: acidity of 0.39 mg KOH/g, viscosity of 2.7 mPa/s, ashes of less than 0.02%, density of 883.7 kg/m3, flash point of 120 ºC, and cetane index of 41. However, it had a high water content.

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1. Introduction

Finding alternative energy sources is currently necessary due to the non-renewability of fossil fuels and their detrimental effects on the environment (Pasha et al., 2021). In this regard, biofuels have emerged as one of the most promising choices in recent years (Mishra & Mohanty, 2022). In terms of environmental, economic, and social sustainability, it is important to keep in mind that biofuels have both benefits and drawbacks. On the one hand, their main drivers globally are the reduction of greenhouse gas (GHG) emissions, energy security, and rural development (Canabarro et al., 2023). On the other hand, there are concerns about rising biofuel production include pressure on food costs, the possibility of increased GHG emissions from both direct and indirect land use change, and the potential of higher food prices.

Second generation raw materials are used to address some of these issues. However, the economic sustainability of some second-generation biofuels is still debatable in the current economic climate, partly because of the low price of oil (Chen et al., 2021). Some of the main benefits of biodiesel produced sustainably include: i) it reduces the life cycle greenhouse gases by an average of 74%; ii) it reduces hydrocarbon emissions by 67%; and iii) it returns 4.56 units of renewable energy for every unit of fossil energy used to produce biodiesel (Canabarro et al., 2023).

One of the widely used types of biofuel made from animal and vegetable fats, is biodiesel, which is regarded as an appealing replacement for fossil diesel. Biodiesel is utilized all over the world in its purest form (100%) denoted by B100 or in blends with petrodiesel as 5% (B5), 20% (B20), and 80% (B80) (Ameen et

al., 2022). It is biodegradable, non-toxic, renewable, and devoid of benzene and sulfur (Knothe & Razon, 2017). These advantages have led to the production of 36 billion liters of biodiesel in 2017 and an estimated 9% rise through 2027 (OECD/FAO, 2018). In Europe, rapeseed and used cooking oil are the main feedstocks for biodiesel production. Argentina, Brazil, and the US also produce significant amounts of biodiesel, primarily from soybeans. Malaysia and Indonesia use palm oil. Biodiesel production more than tripled between 2008 and 2018, from 12 to 41 billion liters (Jarunglumlert et al., 2022).

Reviewing production methods and technologies, or how and what methods are used to optimize the processes that imply the rational use of agricultural inputs, involves analysis of the efficiency in the production of biofuels in Latin America. Forestry, organic waste, and technologies used in the production of first- and second-generation biofuels are among those reviewed (Acharya & Perez, 2020). Trindade et al. (2022) have analyzed in particular the method of turning oil into biodiesel with regard to the effectiveness of the procedure.

Alkali-catalyzed transesterification of fresh vegetable oils is the most popular and financially viable way of producing biodiesel on an industrial scale. However, using fresh edible oils as a feedstock account for more than 80% of the cost of producing biodiesel (Moya et al., 2019). Therefore, using alternate and less expensive sources of lipids, such recovered grease trap waste, is the main approach to lowering the cost of biodiesel manufacturing (Pasha et al., 2021). In this way, the utilization of oily waste products from the fishing industry ties renewable energy production to sustainability.

Large fishing industries have been established in the city of Manta, Ecuador, leading to significant environmental issues (Marn et al., 2015), including wastewater with high BOD levels, fish particles, and foams with oily characteristics from a variety of daily activities (Cedeño et al., 2020). 49% of these residues are made up of oils and fats (Trindade et al., 2022). Medina et al. (2020) classify this waste as of great interest because it is considered an alternative energy source capable of meeting the needs of the industry as such. Oily scums are typically collected in grease traps and placed in metal reservoirs for transfer to landfills.

In the light of these developments, the objective of this study is to assess the impact of physicochemical factors on the efficiency of the process for producing biodiesel from oily byproducts of the fishing industry. The utilization of a product from wastewater discharges during the processing of tuna gives the

research considerable environmental significance, whereby this waste gets transformed into useable material (Mishra & Mohanty, 2022).

The proposed hypothesis focuses on proving whether the influence of temperature, molar ratio, and catalyst concentration increases the performance of the process in the extraction of biodiesel from oily residues. This enables verifying the quality of the biodiesel, so as to contribute to a cleaner production by fishing companies that have committed themselves to protecting the environment through supporting Objective 7 of the 2030 Agenda on Sustainability for the realization of accessible and clean energy.

2. Materials and Methods

This study uses an experimental design to examine the effects of temperature, molar ratio, and catalyst concentration on the biodiesel production performance made from oily waste collected in grease traps at a tuna firm in Manta canton, Manabi province, Ecuador.

According to Lopez et al. (2015), vacuum filtering through a quantitative filter paper no. 40, which can retain up to 25 m suspended particles, must be done if the acid value is less than 1%. It is advised to heat the oil to 80 °C to reduce viscosity.

By using the technique of discrete particle sedimentation and vacuum filtering through a quantitative filter paper no. 40 (capable of keeping suspended solids as small as 25 m), the initial composition of the material entering from the grease traps was separated. The biodiesel was heated to 80 °C before filtering to reduce viscosity (Onur et al*.,* 2018); The extracted oil was then evaluated using parameters such as density, viscosity, acidity index, and saponification index (Jarunglumlert et al*.,* 2022). This allowed the oil to be corrected and its quality to be improved through neutralization, washing, drying, dehydration, and filtering processes (Wang et al*.,* 2022). The following physicochemical analyses needed 1,000 cm^3 (1 liter) of the extracted oil:

- Moisture is determined by weighing the biodiesel after heating it to 105 °C in a stove and then putting it in a desiccator. The difference in weight is used to measure the amount of water or humidity contained in the sample.
- Viscosity: The volume of a liquid flowing by gravity through a calibrated glass capillary viscometer was measured in order to determine the

kinematic viscosity of the products obtained, both transparent and opaque.

- Density at 20°C:

$$\rho \frac{m_{v2} - m_{v1}}{v}$$

ρ = Density

m_{v2} = End mass

m_{v1} = Initial mass

v = Volume

- Acidity index:

$$Acidity\ index = \frac{V * N * 40 * 56{,}11}{P}$$

V = Volume of alkali solution (mL)

N = Normality of the titrated solution

P = Sample weight (g)

40 = Molecular weight of $NaOH$

56,11 = Molecular weight of KOH

- Saponification index:

$$IS = \frac{[(v_1 - v_2)EQ * N]}{m}$$

v_2 = Volume of hydrochloric or hydrogen sulfide used

v_1 = Volume of hydrochloric from blank test

N = Normality of the hydrochloric or hydrogen sulfide (0.5)

m = Mass of sample analyzed

EQ = Chemical equivalent KOH (56.11)

The oil treated in the previous activity was then transesterified using the equipment and methodology suggested by Valencia et al. (2018), which involves using a three-way balloon on a heating system (heating plate) with constant temperature control and a cooling system to prevent the volatilization of methoxide. As a result, the experimental units were processed under the conditions specified in the experimental design (Table 1), obtaining biodiesel samples as a result.

Treatments	Temperature	Molar ratio (MR)	Catalyst concentration (CC)	Amount of oil
T1	60 °C	6:01	0.80%	50 cm^3
T2	60 °C	6:01	1.30%	50 cm^3
T3	60 °C	9:01	0.80%	50 cm^3
T4	60 °C	9:01	1.30%	50 cm^3
T5	80 °C	6:01	0.80%	50 cm^3
T6	80 °C	6:01	1.30%	50 cm^3
T7	80 °C	9:01	0.80%	50 cm^3
T8	80 °C	9:01	1.30%	50 cm^3
				400 cm^3

Table 1. Approach to the experimental design corresponding to a 2^3 factorial arrangement, i.e., three factors (temperature, molar ratio and catalyst concentration) with two levels (high and low).

Subsequently, the biodiesel performance percentages of each treatment were calculated using the following equation (Gheewala et al., 2022). The treatment with the best performance was characterized according to the proposed physical and chemical parameters (Gholami et al., 2020).

$$\%\text{Performance} = \frac{\text{Biodiesel weight (g)}}{\text{Oil sample weight (g)}} \text{X100} \qquad [1]$$

Inferential statistics were employed in the statistical analysis, and InfoStat, R-project[1], and SPSS software were used. This provided a better depiction of the descriptive and visual data. In addition, the analyses of residues, confirmation of

[1] Software used to evaluate the interaction effect and the response Surface.

the assumptions of normality, Shapiro-Wilk test, analysis of variance, and Tukey's test were performed.

3. Results and Discussion

The grease trap oil had the following characteristics: a humidity of 0.40%, a viscosity of 38 mPa/s, and it was within the ASTM-D445 standard's maximum allowable limit of up to 50 mPa/s. The findings of the oil density examination were 0.93 g/ml, and the oil's saponification index was 260.40 mg KOH/g. Additionally, an acidity of 3.63% was obtained. Therefore, it was essential to correct this parameter through the neutralization process, in which NaOH was added in accordance with the dosage established by Arenas et al. (2021), reducing the acidity to 0.97%. In this way, the necessary parameters were met prior to the application of the transesterification process (Table 2).

Parameter	Units	Oil obtained	Maximum limit	Treated oil
Moisture	%	0.40	0.05	0.03
Kinematic viscosity at 40 ºC	mPa/s	38	50	
Density at 20ºC	g/ml	0.93	0.96	
Acidity percentage	%	3.63	0.98	0.977
Acidity index	mg KOH/g	11.08	1.24	2.977
Saponification index	mg KOH/g oil	260.40	Not reported	

Table 2. Physicochemical characterization of the extracted residual oil.

According to the rules of the experimental design, the purified oil was put through the transesterification process at two levels for each variable being studied: temperature (60 and 80 °C), molar ratio (6:1 and 9:1), and catalyst concentration (0.8 and 1.0%). The volume of each treatment was converted to a percentage of the process' performance in relation to the volume of the initial oil, as in equation 1.

In contrast to the study by Kara et al. (2018), which obtained a 99.1% performance from fish residue oil using a 9:1 molar ratio and a 1% catalyst concentration, Figure 1 shows that the T7 treatment (9:1 molar ratio and 0.8% catalyst concentration) presented the best percentage of process performance with 94% in biodiesel extraction. The least successful treatments were discovered

in T2 with 72.67% and T6 with an average of 65.3%. T5 was consistently found with an average of 89.33%; Q3 attained 84%; and the following figures exhibit commonalities between T1, T3, T4 and T8 with their respective averages of 80.67%, 84%, 80%, and 79.33%.

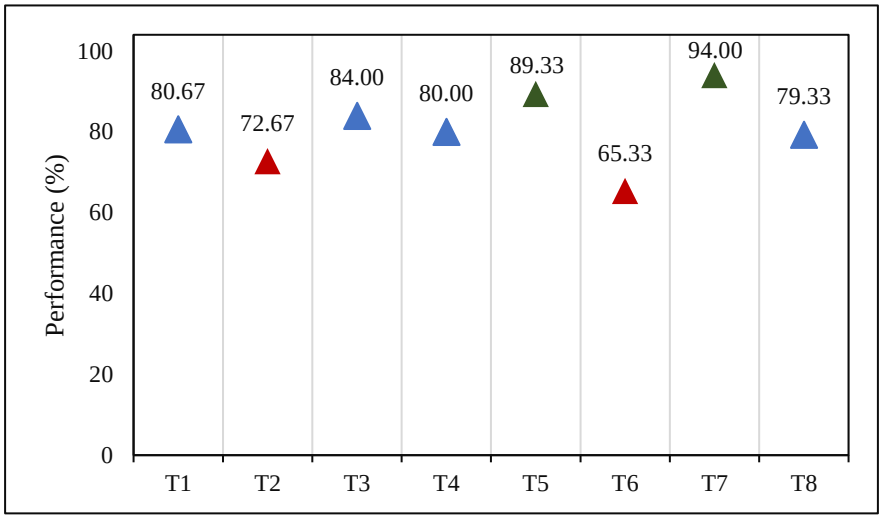

Figure 1. Comparison of the performance obtained according to the treatments.

The major impacts of the variables temperature (A), MR (B), and CC (C) exhibit significance between the levels used, with a p value less than 0.05, according to the analysis of variance of the components employed and their interactions (Table 3).

Temperature and MR (AB), temperature and CC (AC), and MR (alcohol/oil) and CC (BC) interactions were all carried out. It was discovered that the interactions between temperature and MR (AB), temperature and CC (AC), and MR and CC (BC) all showed a p value less than 0.05, presenting statistical significance. The interactions between temperature, MR, and CC (ABC), on the other hand, have a p-value of 0.114, indicating that their interactions do not provide significant means.

F.V	SC	DF	CM	F	P-VALUE
Model	1696	7	242.29	63.2	0.0001
MAIN EFFECTS					
A: Temperature	42.67	1	42.67	11.13	0.0042
B: Alcohol/oil molar ratio	322.67	1	322.67	84.17	0.0001
C: Catalyst concentration	962.67	1	962.67	251.13	0.0001
INTERACTIONS					
AB	24	1	24	6.26	0.0236
AC	266.67	1	266.67	69.57	0.0001
BC	66.67	1	66.67	17.39	0.0007
ABC	10.67	1	10.67	2.78	0.1147
Error	61.33	16	3.83		
Total	1757.33	23			

Table 3. Analysis of variance of the performance of the process.

Figure 2 illustrates the interaction effect between molar ratio (MR) and catalyst concentration (CC), which results in an 89% performance when the CC is low and the MR is high. In his research Avellaneda (2010) came to the conclusion that there must be a balance between the proportions of MR and CC, indicating having acquired a performance of 88.51% when combining MR 9:1 and CC of 0.8%, a figure that is close to 89% performance of the procedure obtained in the current analysis.

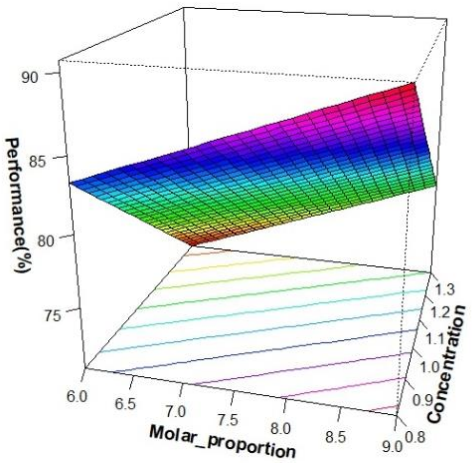

Figure 2. Response surface for biodiesel performance of the process in relation to the CC and MR.

An average performance of 86.7% (Figure 3) was achieved when temperature and MR interacted, emphasizing the high levels of temperature (80 °C) and MR (9:1), comparable to the condition described by Gómez et al. (2022), who attained a performance of 77.89% at 56 °C and a MR of 135:1.

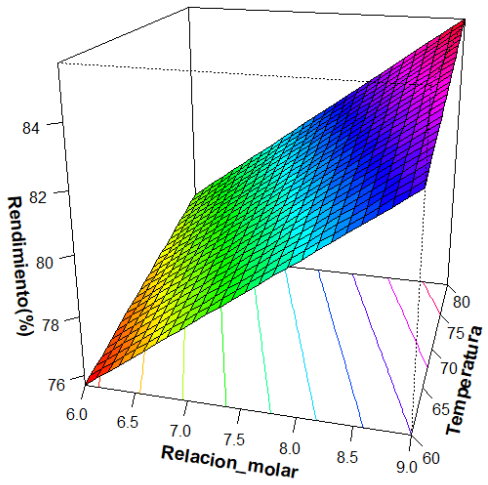

Figure 3. Response surface for process performance in relation to temperature and MR.

In the lowest concentration settings (0.8%), at the highest temperature (80 °C), the temperature and CC interaction effect showed an average performance of 91.67% (Figure 4). Caro et al. (2017) note that it is desirable to apply a CC of less than 1% and attribute this to saponifying materials that are created in excess of a catalyst (NaOH) and causing a deterioration in the performance of the process. Furthermore, Gomez *et al.* (2022) discovered that a temperature rise promotes performance at low catalyst concentrations, while a temperature increase has a detrimental impact on biodiesel performance when employing high catalyst concentrations.

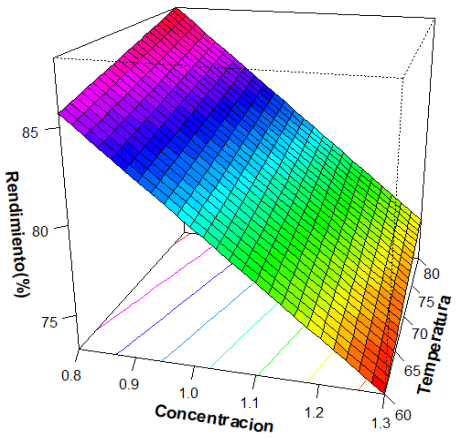

Figure 4. Response surface for process performance in relation to CC and temperature.

The treatment T7 (Temperature 80 °C, 9:1, CC 0.8) provided an average performance of 94.00% throughout the biodiesel extraction process, according to the results of the Tukey test for the treatments examined (Table 4).

TREATMENT	Mean	n	E.E	Group				
				1	2	3	4	5
T6 (1,3.6:1.80)	65.33	3	1.13	A				
T2 (1,3.6:1.60)	72.67	3	1.13		B			
T8 (1,3.9:1.80)	79.33	3	1.13			C		
T4 (1,3.9:1.60)	80.0	3	1.13			C		
T1 (0,8.6:1.60)	80.67	3	1.13			C		
T3 (0,8.9:1.60)	84.0	3	1.13			C	D	
T5 (0,8.6:1.80)	89.33	3	1.13				D	E
T7 (0,8.9:1.80)	94.00	3	1.13					E

Table 4. Tukey's test of the applied treatments.

The treatment T7 (Temperature 80 °C, 9:1, CC 0.8) provided an average performance of 94.00% throughout the biodiesel extraction process, according to the results of the Tukey test for the treatments examined (Table 4).

Additionally, a significant interaction between the factors AB, AC, and CB was discovered (Figure 5) when non-parallel lines were present in their relationship. This resulted in a demonstration of the prevalence of the levels of each factor specified in T7 (80 °C, 9:1, and 08%) and highlighted their greater influence on the efficiency of the biodiesel production process.

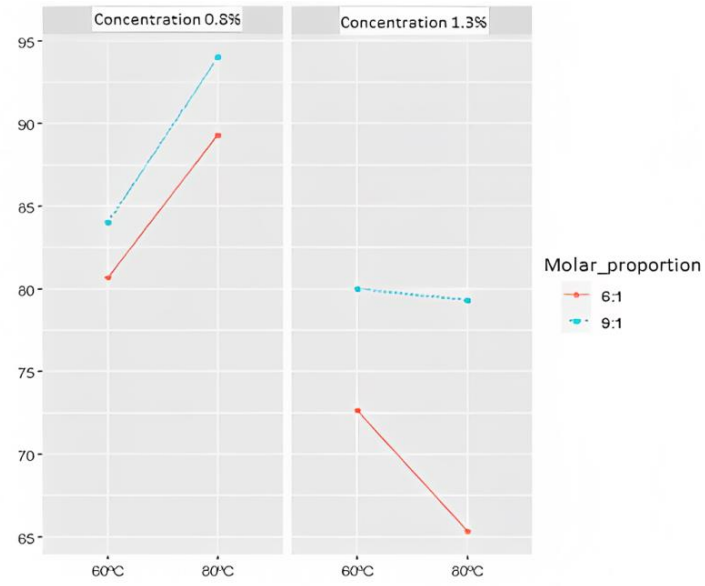

Figure 5. Interaction of the variables used to obtain the performance of the process.

The magnitude of the effects caused by the variables and any potential interactions are expressed in Figure 6, which shows that the CC variable is crucial for determining the percentage of biodiesel extraction. A surplus causes saponification, while a negligible portion results in a conversion deficit (Gomes et al., 2018). Similar results are obtained when the variable C, temperature, and factor B (molar ratio) are combined. Finally, the combination of factors A*B*C.

did not have a bigger impact on the performance percentage than factor A (Temperature).

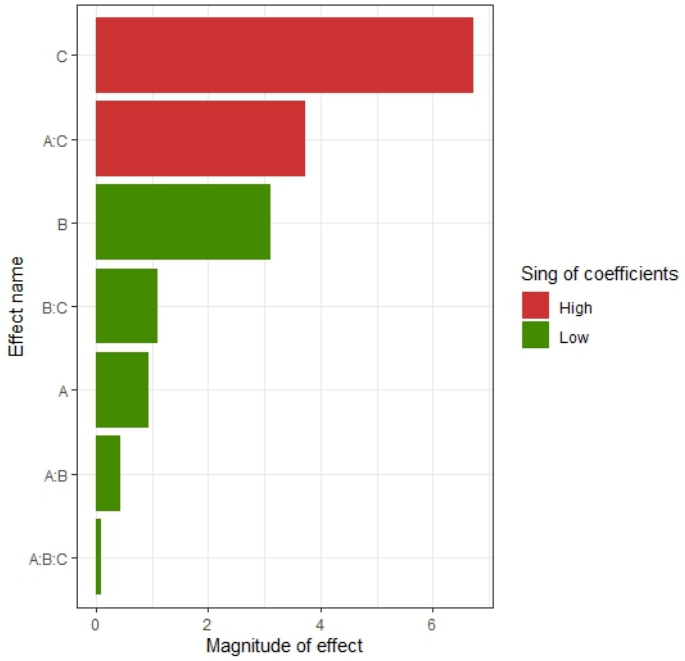

Figure 6. Pareto diagram of process performance. (A: Temperature; B: Molar ratio and C: Catalyst concentration).

The volume of the effect of the catalyst concentration is consistent with that reported by López et al. (2015), who found that the amount of catalyst used affects saponification because of the presence of sodium and its electronegative power. Rahman et al. (2022) also investigated the effect of the amount of catalyst and found that using a concentration close to 0.5% results in a higher performance of biodiesel.

The biodiesel with the greatest performance percentage (T7) is characterized in Table 5, and its density of 883.7 kg/m3 is within the acceptable range for a

biofuel (biodiesel), which is between 860 and 900 kg/m³ (ASTM D6751 and EN14214).

The water content value was 560 mg/kg, which is more than the 500 mg/kg upper limit. Murcia et al. (2013) discovered comparable values of 516 mg/kg in biodiesel made from waste oils. However, they made the point that high levels of water content result in issues with corrosion and the development of microbes in the engine. Brahma et al. (2022) advise utilizing a vacuum evaporation method instead since it allows for higher water extraction without harming the final product.

The findings of Table 5 showed that the extracted biodiesel's acidity index was 0.392 mg KOH/g of oil, complying with the set parameters (which are a maximum of 0.5 mg KOH/g), and that the ash value correlated to 0.02%, meeting the specifications for the manufacturing of biodiesel. Furthermore, the described biodiesel's viscosity was measured at 2.7 mPa/s, which, in accordance with the criteria of Table 5, falls within the permitted range of 3.5 to 5 mPa/s (EN14214).

Parameter	**Unit**	**Biodiesel**	**Specification**	
			LOW	**HIGH**
Density a 15 ºC	kg/m³	883.7	860	900
Ash	%(m/m)	0.015		0.02
Water content	mg/kg	560	--	500
Acidity	%	0.27		
Acidity index	mg KOH/g	0.392	--	0.5
Kinematic viscosity 40 ºC	mPa/s	2.7	3,5	5
Flashpoint	ºC	120	120	--
Cetane index	-	41.62	49	--
Calorific power	MJ/kg	34.4		

Table 5. Physical-chemical parameters of the biodiesel with the best performance.

It was discovered that the extracted biodiesel had a flash point of 120 ºC (ASTMD6751), values that are acceptable for a biodiesel. Moreover, Avellaneda (2010) adds that a biofuel benefits from keeping high levels of flash point.

On the other hand, the biodiesel displayed a cetane index of 41.62, which is below the suggested threshold of 49.00 (ASTM D 976-06). According to

Montenegro et al. (2012), attaining a low cetane index leads to the possibility that excessive noise and ignition problems exist, while Basto et al. (2021) affirm that mixing up to 20% v/v of this additive with diesel is recommended to raise this parameter without affecting the quality metrics.

4. Conclusions

In terms of biodiesel yield, several conclusions were reached as a result of the quantitative research methodologies used to evaluate the impact of physicochemical variables on the effectiveness of the process for producing biodiesel from oily residues in wastewater from fishing industries. The best treatment was found to be achieved with the temperature conditions at 80 °C, alcohol/oil molar ratio of 9:1, and catalyst concentration of 0.8%, presenting a process yield of 94%. The physicochemical characterization of the biodiesel extracted from the grease traps, presented a density (883.7 kg/m^3), ashes (0.015%), and acidity index (0.392). The characterization of the biodiesel extracted from the grease traps, presented a density (883.7 kg/m3), ashes (0.015 %), acidity index (0.392 mg KOH/g), kinematic viscosity (2.7 mPa/S), point of inflammation (120 ºC) and calorific power (34.4 MJ/kg), conditions that meet the specifications for producing biodiesel. Thus, the research hypothesis where the variables Temperature (A), Molar ratio (B) and Catalyst concentration (C) were used for biodiesel extraction demonstrated a significant effect on biodiesel yield.

Thus, this study has shown that it is possible to produce biodiesel from oily residues in wastewater from the fishing industry. The significance of the findings emerges not only through how they relate to and confirm previous research that has already been published, but also through the way they provide a standard for the management of oily waste from fishing enterprises throughout the Ecuadorian Coast.

As a replacement for imported fossil diesel that improves air quality, biodiesel also has economic and environmental advantages (Mizik & Gyarmati, 2021). Oily byproducts from the fishing industry are a free raw material, thereby offering a key component of sustainability within the production process of biodiesel in terms of such features as renewability, biodegradability, and carbon neutrality (Yusoff et al., 2020). Through providing a promising solution to the problem of the management of fishing industry waste, an important contribution can also be made to reducing air pollution and mitigating the greenhouse effect.

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Funds

This research received no specific grant from any funding agency in the public, commercial, or no-profit sectors.

Competing Interests

The authors hereby state that there are no financial or non-financial competing interests.

Citation

Vivas Saltos, H.T., & Cedeño Vargas, M.B. (2023) Fishing industries' oily wastewater biodiesel performance. *Visions for Sustainability*, 20, 7413, 455-474. http://dx.doi.org/10.13135/2384-8677/7413