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To cite this article: Edy Purwanto and Aloisiyus Yuli Widianto 2025 *IOP Conf. Ser.: Earth Environ. Sci.* **1445** 012061

Biodiesel synthesis from palm cooking oil utilising sulfuric acid catalyst in a circular microreactor: Optimisation using response surface method

Edy Purwanto* **and Aloisiyus Yuli Widianto**

[1] Chemical Engineering Department, University of Surabaya, Surabaya, Indonesia

*E-mail: edypurwanto@staff.ubaya.ac.id

Abstract. Biodiesel, a renewable and biodegradable fuel, has gained attention as a viable alternative to traditional petroleum-based diesel. Palm oil is an appealing raw material for biodiesel production due to its high oil yield, availability, and favourable fatty acid profile. Microreactor technology has improved the efficiency and control of chemical reactions, especially in biodiesel production. The key challenge is optimising reaction conditions to maximise Fatty Acid Methyl Ester (FAME) content while reducing the kinematic viscosity of the oil. To address this, the Response Surface Methodology (RSM) was employed. The effects of critical input variables such as catalyst loading, reaction temperature, and oil-to-methanol molar ratio on FAME yield and kinematic viscosity were analysed, and the optimal conditions were identified. Transesterification was carried out using a sulfuric acid catalyst in a circular microreactor, with a central composite design examining three input variables and two output variables. Results showed that catalyst loading, reaction temperature, and oil-to-methanol molar ratio significantly increased FAME content and lowered kinematic viscosity. The optimal conditions were determined to be 4.7% catalyst loading, a temperature of 66.8°C, and an oil-to-methanol molar ratio of 1:10.

1. Introduction

Growing concerns about environmental pollution and the dwindling supply of fossil fuels have sparked interest in researching sustainable and renewable energy sources. Biodiesel, a renewable and biodegradable fuel, has become a promising substitute for traditional petroleum-based diesel [1]. Biodiesel, produced from vegetable oils, animal fats, and used cooking oils, provides benefits such as lower greenhouse gas emissions, biodegradability, and compatibility with current diesel engines [2-6]. Palm oil is particularly appealing among the various feedstocks due to its high oil content, widespread availability, and beneficial fatty acid composition [7,8].

Conventional biodiesel production entails transesterifying triglycerides using an alcohol and a catalyst, typically an acid, base, or enzyme [9]. The choice of catalyst significantly impacts the reaction rate, yield, and purity of the biodiesel produced [10]. Acid catalysts, like sulfuric acid, are incredibly efficient for processing feedstocks with high free fatty acid content, a common characteristic of palm oil [11]. However, the key challenge is optimising reaction conditions to maximise biodiesel yield, specifically Fatty Acid Methyl Ester (FAME) content and process efficiency, while minimising side reactions and reducing catalyst usage.

Microreactor technology has opened new avenues for enhancing the efficiency and control of chemical processes [12]. Channel microreactors provide considerable advantages for biodiesel synthesis due to their high surface-area-to-volume ratio and excellent heat and mass transfer characteristics [12]. These reactors enable precise control over reaction conditions, reduced reaction time, and enhanced safety, making them ideal for optimising complex chemical reactions such as biodiesel production [13,14].

This study investigates biodiesel production from palm cooking oil utilising sulfuric acid as a catalyst within a circular microreactor. The reaction parameters are optimised using Response Surface Methodology (RSM). This statistical technique allows for systematically examining the interactions between multiple variables and identifying optimal conditions. RSM not only tools in optimising the FAME content and kinematic viscosity but also provides valuable insights into the significant effect of multiple input variables on multiple output variables.

However, there is still a lack of research on biodiesel synthesis in a circular microreactor using acid catalysts, particularly the significant effect of input variables on output variables. Therefore, this research aims to contribute to developing efficient and sustainable biodiesel production methods, addressing the Sustainable Development Goal (SDG) 7 issue regarding affordable and clean energy. By leveraging the benefits of microreactor technology and advanced optimisation techniques using RSM, this research seeks to advance the field of biodiesel synthesis and pave the way for its broader adoption as a clean and renewable energy source. Additionally, this research seeks to measure the effects of various input variables-catalyst loading, reaction temperature, and the oil-to-methanol molar ratio on the output variables of FAME content and kinematic viscosity.

2. Materials and methods

2.1 Materials

Palm cooking oil was purchased from a supermarket in Surabaya. Methanol (Merck), sulfuric acid (H_2SO_4) 95-97% (Merck), potassium hydroxide (Merck) and magnesium sulphate anhydrous (RPI) were used without further purification.

2.2 Experimental setup

The transesterification reaction was run in a circular microreactor with ID 1 mm and length 2 m made from PTFE material. The microreactor was placed in a water bath that could maintain the temperature at a desired level. The palm cooking oil was prepared in the first syringe pump, and methanol with the sulfuric acid catalyst in another syringe pump were injected into the circular microreactor through the T junction. This study utilised two syringe pumps (NEMESYS high-pressure syringe pump type NEM-B203–01 B) to maintain a consistent liquid flow throughout the microreactor. These pumps can handle liquids at flow rates ranging from 171.0 nl/min to 825 ml/min, with a maximum pumping pressure of 12 bar for a 100 ml syringe. The two-phase flow pattern was visualised using a High-Speed Digital Camera (CCD HCC-1000) and an SMZ-10 microscope (Nikon), along with image processing software NV 1000 (New Vision Technologies) and a light source (LED lamp). It allows the capture of the slug shape and detects colour changes. The experimental setup is illustrated in Figure 1.

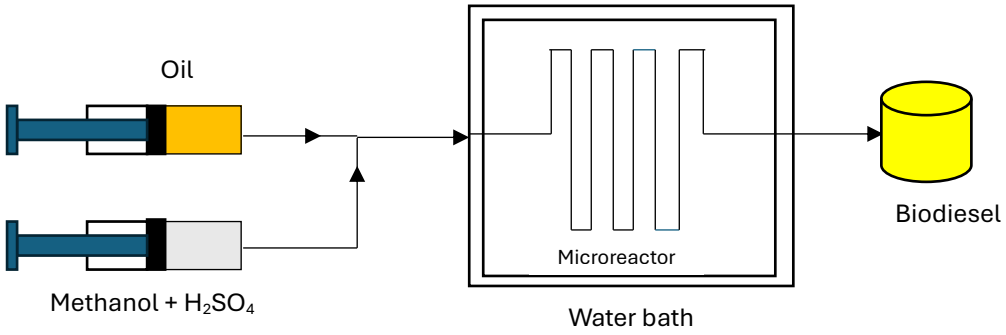


Figure 1. Experimental setup.

2.3 Characterisation of palm cooking oil

The fatty acid composition of palm cooking oil was assessed by converting it to FAME through derivatisation with a sulfuric acid catalyst, and the modified procedure is outlined as follows [15]: 25 g oil, 5 ml sulfuric acid catalyst and 100 ml methanol were added into the round-bottom flask. Then, the mixture was refluxed at 65°C for a 10-h reaction time. Next, the refluxed product was neutralised using potassium hydroxide, extracted with water, and separated into organic and water phases. Next, magnesium sulphate was added to the oil phase and filtrated to acquire water-free oil. The oil phase, which contains FAME, was analysed using Gas Chromatography (GC) to assess the fatty acid composition of palm cooking oil.

2.4 Biodiesel synthesis

The first syringe pump was designated for the oil phase containing palm cooking oil, while the second syringe pump was set up with an aqueous phase consisting of a mixture of methanol and sulfuric acid catalyst. The circular microreactor used for the transesterification reaction was placed in a water bath to control the reaction temperature. Each reactant in the syringe was then pumped into the microreactor, and the reaction temperature was maintained as expected, following the experimental design mentioned in Table 3. The methanol and sulfuric acid flow rate mixture was maintained at 0.0334 ml/min. Conversely, the oil flow rate was adjusted within the range of 0.0231 to 0.0658 ml/min to achieve the desired molar ratio of oil to methanol, as outlined in the experimental design presented in Table 3. The oil and methanol flow rates applied at that range result in a stable slug flow pattern with a regular liquid velocity along the channel. Biodiesel synthesis is carried out under a slug flow pattern. The reaction product was collected, neutralised and then cooled. Water was introduced into the separating funnel to extract methanol from the oil phase, and the mixture was decanted to separate the oil phase (FAME) from the water-soluble components. The FAME was subsequently washed with warm water until the wash water reached a neutral pH. Magnesium sulphate anhydrous was added to the FAME oil to separate water from FAME and then filtrated. The FAME product was analysed using gas chromatography, and the FAME content was determined using equation 1 [16]:

$$\%FAME = \frac{area\ all\ FAME}{area\ of\ all\ reference} x \frac{mass\ of\ reference}{mass\ of\ biodiesel\ sample} \quad (1)$$

The kinematic viscosity was measured using the viscometric method.

2.5 Experimental design and optimisation of biodiesel synthesis

A full factorial central composite design (CCD) was employed, and the total number of experiments can be calculated using the equation $2^n + 2n + n_0$ [17]:

where: n = the number of input variables
2^n = factorial design
2n = star point
n_0 = the number of replications at the central value.

The optimisation was conducted for three input variables (catalyst loading, reaction temperature, and oil-to-methanol molar ratio), designated as X_1, X_2 and X_3 across five levels (-1.682,-1,0,1,1.682) according to the equation:

$$X_i = \frac{x_i - x_0}{\Delta x_i} \quad (2)$$

Where: X_i = the coded value of the input variable
x_i = the actual value of the input variable
x_0 = the actual value of the input variable at the central value
Δx_i = the interval value

The central value (n_0) for biodiesel synthesis was defined by a catalyst loading of 3%, a temperature of 50°C, and an oil-to-methanol molar ratio of 1:7. The range and levels of the input variables are displayed in Table 1.

Table 1. The range and levels of variables.

Input variables	Coded values	Levels				
		(-1.682)	-1	0	1	1.682
Catalyst loading (%)	X_1	1.3	2	3	4	4.7
Reaction temperature (°C)	X_2	33.2	40	50	60	66.8
Molar ratio oil to methanol	X_3	1:3.6	1:5	1:7	1:9	1:10.4

2.6 Statistical analysis

The relationship between the experimental data variables (outputs) and the input variables in Table 3, expressed in coded values, was modelled using a second-order polynomial, as shown in Equation 3 [18]:

$$Y_n = \beta_0 + \sum_{i=1}^{n} \beta_i X_i + \sum_{i=1}^{n} \sum_{j=1}^{i-1} \beta_{ij} X_i X_j \quad (3)$$

Where Y_n is the output variables
β_i and β_{ij} are model coefficients.

For the effect of three input variables on output variables, equation (3) is further explained as:

$$Y_n = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \beta_{12} X_{12} + \beta_{13} X_{13} + \beta_{23} X_{23} \quad (4)$$

Where $Y_n (n = 1 - 2)$ is the output variables (FAME content and kinematic viscosity); β_0 is constant; β_1, β_2 and β_3 are the linear coefficients; β_{11}, β_{22} and β_{33} represent the quadratic

coefficients; β_{12}, β_{13} and β_{23} are the interaction coefficients. Minitab software was utilised to generate the response surface regression within the Analysis of Variance (ANOVA) package.

3. Results and discussions

3.1 Palm cooking oil characteristics

The fatty acid composition of palm cooking oil was analysed using the derivatisation method. Triglycerides in the oil were reacted with methanol to produce methyl ester using an acid catalyst, allowing a complete reaction. The Fatty Acid Methyl Ester (FAME) products were subsequently analysed using Gas Chromatography (GC), and the resulting FAME composition was converted into fatty acid composition. Table 2 displays the fatty acid composition of palm cooking oil determined through the derivatisation method.

Table 2. Composition of fatty acids in palm cooking oil.

Fatty acid	Composition (%)
Lauric acid	0.65
Miristic acid	1.35
Palmitic acid	38.27
Oleic acid	41.04
Linoleic acid	18.69

3.2 Statistical analysis of response surface method

A total of 20 experiments were carried out for all specified points according to the variable ranges and levels outlined in Table 1, along with the experimental design presented in Table 3. The experimental points design was conducted in a randomised order, and the results for the two output variables (FAME content and kinematic viscosity) are also shown in Table 3.

Table 3. The Experimental design and output variables.

Exp No	Catalyst loading (%)	Reaction Temperature (°C)	Molar ratio oil to methanol	FAME content (%)	Kinematic viscosity (cSt)
	X_1	X_2	X_3	Y_1	Y_2
1	1 (4)	1 (60)	-1 (1:5)	55.78	15.17
2	1 (4)	-1 (40)	1 (1:9)	40.94	20.58
3	0 (3)	0 (50)	0 (1:7)	40.58	20.91
4	-1 (2)	1 (60)	1 (1:9)	42.78	18.85
5	0 (3)	0 (50)	0 (1:7)	41.35	20.16
6	-1 (2)	-1 (40)	-1 (1:5)	11.64	29.78
7	0 (3)	0 (50)	0 (1:7)	42.04	19.43
8	1.682 (4.7)	0 (50)	0 (1:7)	58.41	14.90
9	0 (3)	0 (50)	0 (1:7)	40.37	19.73
10	0 (3)	0 (50)	0 (1:7)	41.95	19.07
11	0 (3)	0 (50)	-1.682 (1:3.6)	28.46	24.22
12	1 (4)	-1 (40)	-1 (1:5)	33.51	22.84
13	0 (3)	1.682 (66.8)	0 (1:7)	49.05	17.81
14	-1 (2)	-1 (40)	1 (1:9)	23.22	25.95
15	-1.682 (1.3)	0 (50)	0 (1:7)	15.93	28.77
16	0 (3)	-1.682 (33.2)	0 (1:7)	20.96	25.68
17	-1 (2)	1 (60)	-1 (1:5)	29.98	23.72

Exp No	Catalyst loading (%)	Reaction Temperature (°C)	Molar ratio oil to methanol	FAME content (%)	Kinematic viscosity (cSt)
	X_1	X_2	X_3	Y_1	Y_2
18	1 (4)	1 (60)	1 (1:9)	64.41	12.21
19	0 (3)	0 (50)	1.682 (1:10.4)	46.37	18.29
20	0 (3)	0 (50)	0 (1:7)	45.89	18.45

The FAME content (Y_1) and kinematic viscosity (Y_2) were measured during the transesterification reaction in the microreactor to establish their correlation with the respective input variables: catalyst loading (X_1), reaction temperature (X_2) and oil-to-methanol molar ratio (X_3). Response surface regression was conducted using Minitab software to assess the relationship between the output variables Y_1 and Y_2 and the input variables (X_1, X_2, X_3). Table 4 represents the obtained regression statistics, whereas Table 5 provides the analysis of variance (ANOVA). The data correlation value (R^2) of 0.985 for FAME content and 0.969 for kinematic viscosity demonstrated a strong fit for the model in illustrating the relationship between the input and output variables.

Table 4. Regression statistics for FAME content and kinematic viscosity.

Regression statistics	FAME content (Y_1)	Kinematic viscosity (Y_2)
R^2	0.985	0.969
Adjusted R^2	0.972	0.941
Predicted R^2	0.923	0.823
Standard error	2.310	1.099

Table 5. Analysis of Variance (ANOVA) for the model.

Parameters	FAME content (Y_1)	Kinematic viscosity (Y_2)
DF	9	9
Adjusted SS	3575.13	378.145
Adjusted MS	397.24	42.016
F-value	74.47	34.81
P-value	0.000	0.000

Table 6. Significance of regression coefficients for FAME content (Y_1).

	Coefficients	Standard error	T-value	P-value
Constant	41.979	0.942	44.57	<0.001
X_1	9.584	0.625	15.33	<0.001[a]
X_2	11.603	0.625	18.57	<0.001[a]
X_3	5.167	0.625	8.27	<0.001[a]
X_1^2	-2.149	0.608	-3.53	0.005[b]
X_2^2	-1.384	0.608	-2.27	0.046[c]
X_3^2	-1.297	0.608	-2.13	0.059
X_1X_2	0.980	0.817	1.20	0.258
X_1X_3	0.303	0.817	0.37	0.719
X_2X_3	-1.040	0.817	-1.27	0.232

[a] significant at 0.1% ($p<0.001$)
[b] significant at 1.0% ($p<0.01$)
[c] significant at 0.5% ($p<0.05$)

Table 7. Significance of regression coefficients for kinematic viscosity (Y_2).

	Coefficients	Standard error	T-value	P-value
Constant	19.644	0.448	43.84	< 0.001
X_1	-3.107	0.297	-10.45	<0.001[a]
X_2	-3.722	0.297	-12.52	<0.001[a]
X_3	-1.750	0.297	-5.89	<0.001[a]
X_1^2	0.624	0.289	2.16	0.056
X_2^2	0.656	0.289	2.27	0.047[c]
X_3^2	0.451	0.289	1.56	0.150
X_1X_2	-0.360	0.388	-0.93	0.376
X_1X_3	-0.217	0.388	-0.56	0.588
X_2X_3	0.435	0.388	1.12	0.289

[a] significant at 0.1% (p<0.001)
[b] significant at 1.0% (p<0.01)
[c] significant at 0.5% (p<0.05)

The elevated correlation values (R^2) for the data outputs indicated a strong alignment between the model and the experimental data. Minitab software was employed to analyse the data and visualise the results using surface plots.

3.3 Influence of catalyst loading, temperature, and molar ratio on FAME content

A second-order polynomial model was fitted to the CCD experimental data using response surface regression in Minitab software. The effect of three input variables (catalyst loading, temperature and molar ratio on FAME content (%) in coded values utilising the response surface method is represented by equation 5 below:

$$Y_1 = 41.979 + 9.584X_1 + 11.603X_2 + 5.167X_3 - 2.149X_1^2 - 1.384X_2^2 - 1.297X_3^2 + 0.980X_1X_2 + 0.303X_1X_3 - 1.040X_2X_3 \quad (5)$$

Y_1 (FAME content in %) is the output variable, and X_1, X_2 and X_3 are the input variables (catalyst loading, temperature and molar ratio) in coded values. Figure 2 illustrates the interaction effects of the input variables on the output FAME content. Each plot demonstrates how the interaction of input variables affects the increase in FAME content as the output while keeping the other variables at their central values. Figure 2A illustrates the impact of both catalyst loading and reaction temperature. At a 1:7 molar ratio of oil to methanol, the FAME content of the oil increases more rapidly with rising reaction temperature than with increased catalyst loading. Figure 2B shows a similar effect of catalyst loading and the molar ratio of oil to methanol on the increasing FAME content achieved at a reaction temperature of 50°C, with catalyst loading having a more significant impact than the molar ratio. Figure 2C illustrates the combined effect of reaction temperature and the molar ratio of oil to methanol (at 3% catalyst loading) on FAME content. Both input variables similarly affect the increasing amount of FAME in biodiesel.

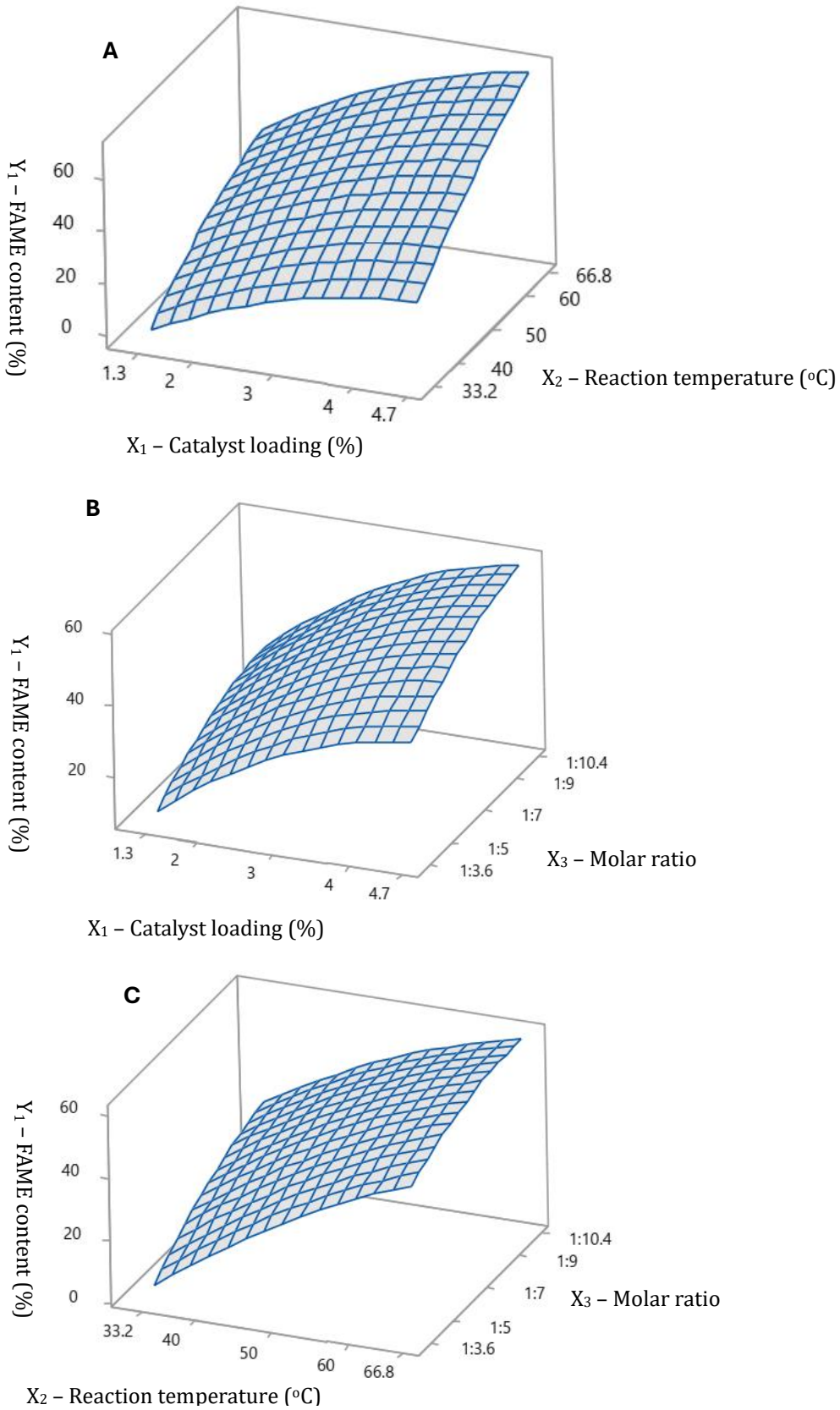


Figure 2. Interactive effect of input variables on FAME content.

Overall, the results demonstrate that the FAME content in biodiesel significantly increases linearly with higher catalyst loading, reaction temperature, and molar ratio of oil to methanol, showing statistical significance at $p<0.001$ (Table 6). The most significant effect of input variables on FAME content is reaction temperature. These input variables (catalyst loading, reaction temperature, and molar ratio) positively impact FAME content. Triglycerides made of fatty acids are transformed into methyl esters in the microreactor via the transesterification reaction, as evidenced by the increasing amount of FAME [19]. There are three steps for the conversion of triglyceride to FAME; follow the reaction mechanism below [20]:

$$\text{TGC} + \text{Me} \leftrightarrow \text{DGC} + \text{ME}$$
$$\text{DGC} + \text{Me} \leftrightarrow \text{MGC} + \text{ME}$$
$$\text{MGC} + \text{Me} \leftrightarrow \text{GLC} + \text{ME}$$

where: TGC = Triglyceride; Me = Methanol; DGC = Diglyceride; MGC = Monoglyceride;
GLC = Glycerol; ME = Methyl Ester

Figure 3 presents the chromatogram of the FAME profiles obtained from this study.

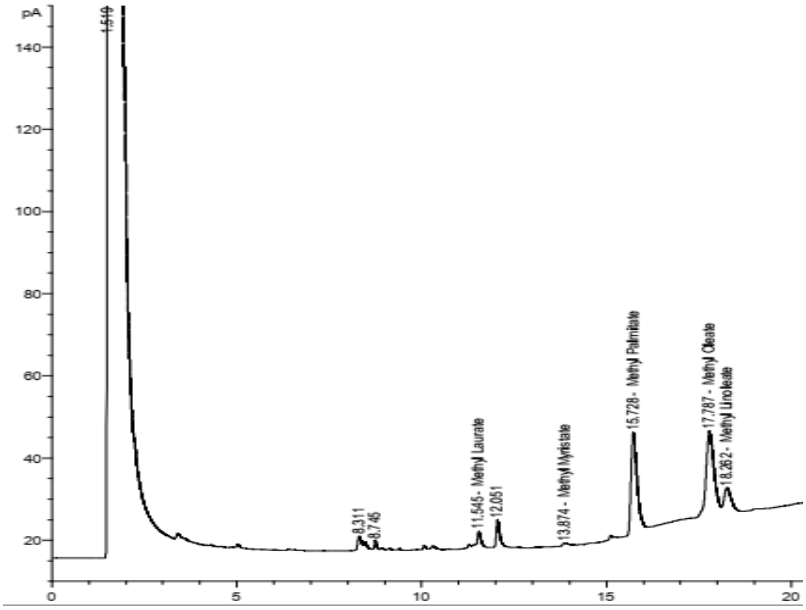


Figure 3. Chromatogram of FAME.

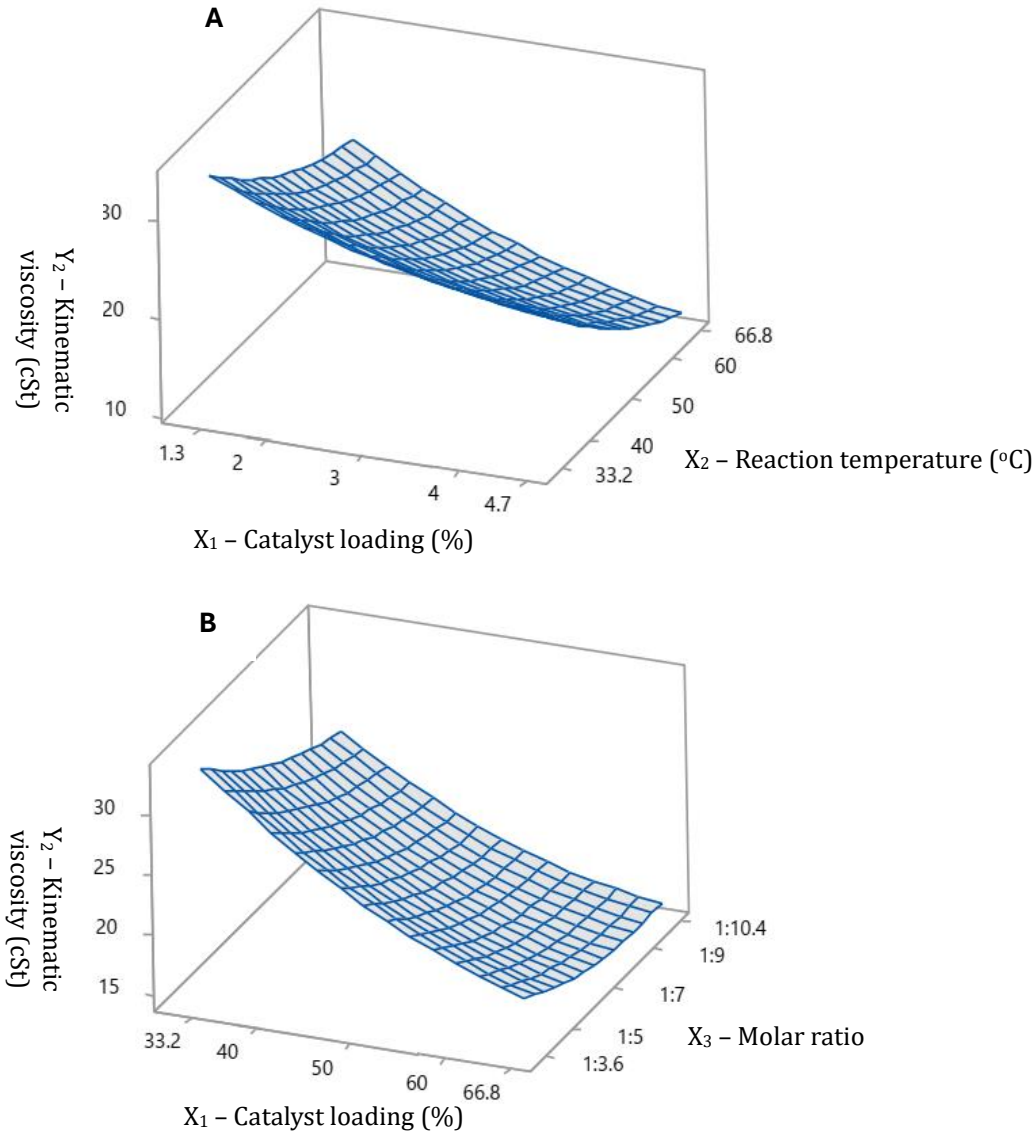
3.4 Influence of catalyst loading, temperature and molar ratio on kinematic viscosity

The correlation equation that relates kinematic viscosity to the three input variables (catalyst loading, reaction temperature, and molar ratio of oil to methanol) in coded units is shown below:

$$Y_2 = 19.644 - 3.107X_1 - 3.722X_2 - 1.750X_3 + 0.624X_1^2 + 0.656X_2^2 + 0.451X_3^2 - 0.360X_1X_2 - 0.217X_1X_3 + 0.435X_2X_3 \quad (6)$$

The results indicate that the kinematic viscosity of biodiesel is a quadratic function of reaction temperature (significant at $p<0.05$) and a linear function of both catalyst loading and the molar ratio of oil to methanol (significant at $p<0.001$), as shown in Table 7.

Figure 4 illustrates the interaction effects of the input variables on kinematic viscosity. Specifically, Figure 4A depicts the combined effects of catalyst loading and reaction temperature on kinematic viscosity. The plot indicates that both catalyst loading and reaction temperature significantly influence the reduction of biodiesel kinematic viscosity. The interaction between catalyst loading and the molar ratio of oil to methanol is presented in Figure 4B. It is also observed that kinematic viscosity decreases significantly when both factors are increased. Figure 4C illustrates how the interaction between the reaction temperature and the oil-to-methanol molar ratio influences the kinematic viscosity. The results demonstrated that increasing both the reaction temperature and the molar ratio of oil to methanol led to a substantial decrease in kinematic viscosity. It was observed that higher levels of catalyst loading, reaction temperature, and molar ratio of oil to methanol negatively impacted the output variable, resulting in a reduction in kinematic viscosity.



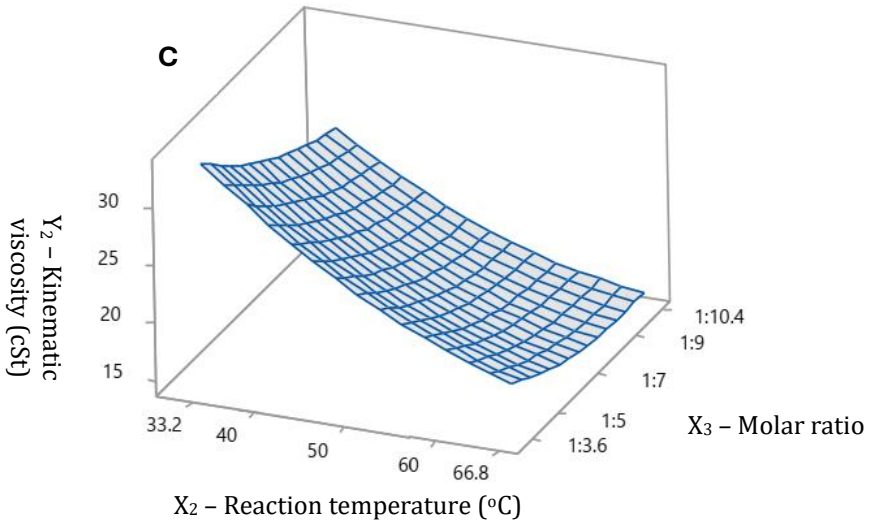


Figure 4. Interactive effect of input variables on kinematic viscosity.

Kinematic viscosity is one of the critical factors for biodiesel properties in determining the quality and performance of biodiesel as a fuel. The aim of transforming palm oil into FAME is to lower the kinematic viscosity of the original oil, producing biodiesel that can be used in diesel engines [21]. The experimental results in Table 3 show that the obtained biodiesel does not satisfy the kinematic viscosity standard for biodiesel in Indonesia, which should be in the range of 2.3 – 6.0 cSt [22]. Higher FAME content will result in lower kinematic viscosity of biodiesel. The residence time of reactants needs to increase by utilising a longer microreactor.

The optimum condition was achieved using a response optimiser in Minitab and obtained by maximising the FAME content point and minimising the kinematic viscosity point. The optimal conditions were identified with a catalyst loading of 1.682, a reaction temperature of 1.682, and a molar ratio 1.512, represented as coded values. The actual values of the input variables at the optimal conditions for the range variables in this study were derived by converting the coded values to actual values using Equation 2, resulting in a catalyst loading of 4.7%, a reaction temperature of 66.8 °C, and a molar ratio of oil to methanol of 1:10.0.

4. Conclusion

The optimisation of biodiesel production using a microchannel reactor was conducted through the response surface method, focusing on the impact of three variables: catalyst loading, reaction temperature, and the molar ratio of oil to methanol. The response surface method is a very efficient way to optimise as it reduces the number of experiments, saves time, and is highly efficient. The findings indicated that higher catalyst loading, longer reaction temperature, and a higher molar ratio of oil to methanol led to increased FAME content and a detrimental decrease in the kinematic viscosity of the biodiesel. These three input variables significantly affect the obtained FAME and kinematic viscosity value. The optimal conditions for biodiesel synthesis using a circular microreactor were achieved with a catalyst loading of 4.7%, a temperature of 66.8 °C, and a molar ratio of oil to methanol of 1:10.0.

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ISSN 1755-1307

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275

VOLUME 275 – 2019

3–2 October 2018
Ancona, Italy

EDITOR
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To cite this article: 2020 *IOP Conf. Ser.: Earth Environ. Sci.* **443** 011003

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Table of contents for issue 1, volume 1445, IOP Conference Series: Earth and Environmental Science

Volume 1445

2025

← Previous issue Next issue →

6th International Conference on Natural Resources and Technology 2024 (ICONART 2024) 27/08/2024 - 27/08/2024 Medan, Indonesia

Accepted papers received: 07 January 2025
Published online: 07 February 2025

Open all abstracts

Preface

OPEN ACCESS 011001
Preface
View article PDF

OPEN ACCESS 011002
Peer Review Statement
View article PDF

Food Science and Food Technology

OPEN ACCESS 012001
Anti-diabetic compounds from *Physalis angulate* L. (ciplukan) and *Andrographis paniculate*: A review
Sammi Chen, Angela Kristiana and Steven Suryoprabowo
View article PDF

OPEN ACCESS 012002
Utilization of sacha inchi oil as an alternative to fish oil: A review
Gabriella Renata and Steven Suryoprabowo
View article PDF

OPEN ACCESS 012003
Improving nutritional and functional properties of sorghum flour: A review

Jane Puteri and Tina Nurkhoeriyati

View article PDF

OPEN ACCESS 012004

Drying technologies utilized to preserve persimmon fruits (*Diospyros kaki* L.): A review

Friska Nathania and Tina Nurkhoeriyati

View article PDF

OPEN ACCESS 012005

Microbial profile of agar-supplemented tempeh: a strategy towards enhancing the values of tempeh as a functional food

Reggie Surya, Kanta Petsong, Andreas Romulo, David Nugroho, Felicia Tedjakusuma and Olifia Rombot

View article PDF

OPEN ACCESS 012006

Supplementation of dried *Porphyra* alga (nori) improves antioxidant activity and isoflavone bioavailability of tempeh

Reggie Surya, Felicia Tedjakusuma, Olifia Rombot and David Nugroho

View article PDF

OPEN ACCESS 012007

Physicochemical and sensory evaluation of snack bars with isomalto-oligosaccharides as a honey substitute

Ryan Reynardo Lie, Felix Widodo, Thitipong Phothisoot, Teeradate Kongpichitchoke and Diana Lo

View article PDF

OPEN ACCESS 012008

Utilization of non-timber forest products *Arenga pinnata* as a natural food source

Siti Latifah, Khaira Amalia Fachrudin, Kansih Sri Hartini, OK Hasnanda Syahputra, Zahrul Ulum, Luthan Ariel Doufan Sihombing, Mia Amelia, Farhan Aziz, Josua Nainggolan and Muhammad Raihan Hawari

View article PDF

OPEN ACCESS 012009

Consumers' awareness and perception of the potential risks of minimally processed food consumption

Ricky Fenton and Ervina Ervina

View article PDF

OPEN ACCESS 012010

Sensory and textural properties of gluten-free muffins made from taro (*Colocasia esculenta*), banana (*Musa acuminata*) and sweet potato (*Ipomea batatas* L.) flours

Ervina Ervina and Nathan Efata Imannuel

View article PDF

OPEN ACCESS 012011

Optimization of oat-based ready-to-drink with tropical fruit flavors development using sensory approach

Gelvin and Nur Fathonah Sadek

View article PDF

OPEN ACCESS 012012

Distribution of sukun (*Artocarpus Communis* Forst) in conjunction with altitude and type of edaphic in North Sumatra, Indonesia

Budi Utomo, Maesy Lerina, Emmy Harso Kartadinata and Lily Fauzia

View article PDF

OPEN ACCESS 012013

Quality evaluation of the combination of Porang flour and Telang flower extract as natural preservatives and antioxidants in extending the shelf of wet noodles

Elimasni, Rizky Yudha Pratama, Isnaini Nurwahyuni and Syamsuardi

View article PDF

OPEN ACCESS 012014

Effect of purple sweet potato paste (*Ipomoea batatas* L.) addition on yoghurt quality

Elisa Julianti, Mimi Nurminah and Beatrice Nathania

View article PDF

OPEN ACCESS 012015

Isolation fragment of pi-d2 as gene resistance to blas disease in Siporang Rice (*Oryza sativa* L.) From Sipirok, North Sumatera

Saleha Hannum, Suci Rahayu and Oktasan Jaya Sihombing

View article PDF

OPEN ACCESS 012016

The potential of eucalyptus leaves from industrial plantation forest logging waste as a raw material for essential oils in PT. Toba Pulp Lestari Tbk

Agus Purwoko, Simon Sidabukke, Rita Kartika Sari, Khairanti Liandari and Ulil Amri Daulay

View article PDF

OPEN ACCESS 012017

The honey of *Trigona biroi* as a bioindicator for detecting toward green city environmental pollution levels

Budiaman, Sitti Nuraeni, Iswara Gautama, Andi Asikin Muchtar and Berlin Rara Andi Eda

View article PDF

Natural Science and Natural Product

OPEN ACCESS 012018

Analysis of forest area management development strategy at UPT Bukit Barisan Grand Forest Park, Indonesia

Leo Bilyanto Sembiring, Samsuri, Bejo Slamet, Siti Latifah and Ma'rifatin Zahrah

View article PDF

OPEN ACCESS 012019

Simulation of subgrade layer model at laboratory modified compaction works in determining CBR value on stabilized expansive soil

Putera Agung Maha Agung, Sutikno, Muhammad Fathur Rouf Hasan, Aida Nurfitria, Agung Sedayu, Zaindra Fakhri Salim and Adi Susilo

View article PDF

OPEN ACCESS 012020

Quality analysis of soap noodles from palm fatty acid distillate-refined bleached deodorized palm stearin blend

Muhammad Ravi Yudiansyah, Rosdanelli Hasibuan and Rondang Tambun

View article PDF

OPEN ACCESS 012021

Analysis of redesigning 3 axis motor control using arduino mega on a CNC milling machine unit

Ahmad Yunus Nasution, Muhammad Ibrahim Syah, Muhammad Sabri, Tulus Burhanuddin Sitorus and Nur Cholis

View article PDF

OPEN ACCESS 012022

Estimation of biomass and carbon storage of *Pinus merkusii* for ecological sustainability at the Sipinsur Geosite Ecotourism

Siti Latifah, Khaira Amalia Fachrudin, Suri Fadhilla, Dodik Ridho Nurrochmat and Anggiat Simanjuntak

View article PDF

OPEN ACCESS 012023

Natural dyes of Purun crafts materials and their effect on fibre dimension and derivative dimensions

Ridwanti Batubara, Shafira Chairunnisa Chery, Arif Nuryawan, Nisa Inayah Amalasari, Iwan Risnasari, Harisyah Manurung, Mohammad Basyuni, Oding Affandi, Luthfi Hakim and Ina Winarni

View article PDF

OPEN ACCESS 012024

Antimicrobial and antioxidant properties of the lichens *Coccocarpia palmicola, Parmotrema clavuliferum* and *Parmotrema tinctorum*

Oky Kusuma Atni, Erman Munir, Etti Sartina Siregar and M. N. Saleh

View article PDF

OPEN ACCESS 012025

The influence of applying local wisdom in sustainable differentiation strategy to achieve competitive advantage in the tourism industry

Ratih Pratiwi, Muhtar, Muhammad Asril Arilaha, Arafat and Yerrynaldo Loppies

View article PDF

OPEN ACCESS 012026

Utilization of kepok banana starch bioplastic spoons: effects of areca nut peel cellulose and glycerol additions

Nisaul Fadilah Dalimunthe, Thiodorus Marvin Tjandra, Evelyn Damayanti Ambarita, Lydia Esterlita Barus, M. Thoriq Al Fath and Rivaldi Sidabutar

View article PDF

OPEN ACCESS 012027

Characteristics of red bean ice cream with the addition of VCO (Virgin Coconut Oil) using hybrid coconut (Khina 2) from local indonesian resources to increase medium chain fatty acids which is good for body immune

Mimi Nurminah, Khoirun Nisa and Elisa Julianti

View article PDF

OPEN ACCESS 012028

Optimizing natural resource utilization: A case of physical and monetary balance of pine resin in forest farmer group

Makkarennu, Sastra Jassen Ruli and Irnasari

View article PDF

OPEN ACCESS 012029

Design of system monitoring discharge flow and volume hydrogen gas based an Internet of Things (IoT) on Electrolyzer

Lukman Hakim and Delima Waruwu

View article PDF

OPEN ACCESS 012030

Morphology of palm sugar trees (*Arenga pinnata*) at the various topography in South Sulawesi

Syahidah, Andang Suryana Soma, Makkarennu, Ira Taskirawati, Chairil, Muhammad Fagil Akbar, Sulkifli, Nur Rezy Parasita, Muh. Ilham Deradjat, Aufar Rifky Utama *et al*

View article PDF

OPEN ACCESS 012031

Regenerative agriculture based Good Agricultural Practices (GAP) for soil health in arabica coffee in North Sumatra

Silvia Nora, Retna Astuti Kuswardani, Surip Mawardi and Aisar Novita

View article PDF

OPEN ACCESS 012032

The Influence of Cinnamon Powder from *Cinnamomum burmannii* bark on quality and storage period of liquid sugar palm *(Arenga longipes* Mogea*)*

Harisyah Manurung, Luthfi Hakim, Ridwanti Batubara, Mhd. Alvi Syahputra and Yunida Syafriani Lubis

View article PDF

OPEN ACCESS 012033

Productivity of Aren (*Arenga longipes* Mogea) in Rumah Sumbul Village, Sibolangit District, Deli Serdang Regency

Harisyah Manurung, Ridwanti Batubara and Romaulina Br Purba

View article PDF

OPEN ACCESS 012034

Bee Feed Diversity at Flora Nauli Beekeepers, Pematang Siantar, North Sumatra Province

Oding Affandi, Lili Permatasari, Ridwanti Batubara and Mariah Ulfa

View article PDF

OPEN ACCESS 012035

Plant therapy to improve mental health of older people in Nangbelawan Village, Karo Regency, Indonesia

Floren Br Barus, Husni Thamrin, Malida Putri, Fajar Utama Ritonga, Agus Suriadi, Mia Aulina Lubis and Izzah Dienillah Saragih

View article PDF

OPEN ACCESS 012036

Carbon tax policy in achieving Indonesia's nationally determined contribution target year 2030

Muhammad Husni Thamrin, Faiz Albar Nasution and Deden Nuryadin

View article PDF

OPEN ACCESS 012037

Analysis of the impact of halal tourism policy on the development of natural resources and the economy of local communities in the Lake Toba Region

Victor Lumbanraja, Muhammad Imanuddin Kandias Saraan and Moulita

View article PDF

OPEN ACCESS 012038

Antifungal activity of *Cinnamomum iners* bark extract against *Schizophyllum commune*

Astuti Arif, Jusniar Bachtiar and Syahidah

View article PDF

OPEN ACCESS 012039

Potential of Malacca (*Phyllanthus emblica* L.) wood extracts as natural dyes for textiles

Iwan Risnasari, Khaira Amalia Fachrudin, Romario Sitinjak, Fatimah Rangkuti and Agus Handoyo

View article PDF

OPEN ACCESS 012040

Histosol soil respiration rate study: measurement and implications in Humbang Hasundutan Regency

Sarah Patumona Manalu, Yasmine Anggia Sari and Ade Citra Nadhira

View article PDF

OPEN ACCESS 012041

Carbon negativity of black locust and poplar plantation in different management systems in Hungary

Budi Mulyana, András Polgár and Andrea Vityi

View article PDF

OPEN ACCESS 012042

The Effect of vegetation density on infiltration rate in the Suso Watershed

Wahyuni and Warda Wiyana Habir

View article PDF

OPEN ACCESS 012043

Reduction of microplastics through waste management: Evaluation of water quality and health risks in the communication policy of the ministry of environment and forestry

Dewi Kurniawati, Maulana Andinata Dalimunthe, Syukur Kholil, Aulia Rahma Ritonga and

Teguh Agum Pratama

View article PDF

OPEN ACCESS 012044

Discovering the potential of endophytic bacteria isolated from *Styrax paralleloneurus* as IAA production

Arida Susilowati, Rumella Simarmata, Yeni Khairina, Firda Oktarina, Wina Nur Saputri, Siti Halimah Larekeng, Tiwit Widowati, Deni Elfiati and Margaretta Christita

View article PDF

OPEN ACCESS 012045

Characteristics of wood adhesive made of gambier from Dairi and Pakpak Bharat, North Sumatra

Tito Sucipto and Toba Wijaya Lumbantoruan

View article PDF

OPEN ACCESS 012046

The economic value and marketing chain of gambir (*Uncaria gambir* Roxb) in Pakpak Bharat Regency, North Sumatra

Tito Sucipto, Priskian Siboro, Muhammad Afif Alfaritsi and Apri Heri Iswanto

View article PDF

OPEN ACCESS 012047

Mitigating food waste and household waste management: The potential for redistributing surplus food in the policy communication of Medan City government

Feni Khairifa, Syukur Kholil, Abdi Mubarak Syam and Naqil Sayyaf Al-Mujahid

View article PDF

OPEN ACCESS 012048

Growth of trembesi (*Samanea saman*) seedlings on waste growing media which modified with organic matter

Alfan Gunawan Ahmad, William A Sagala and Moehar M Harahap

View article PDF

OPEN ACCESS 012049

Analysis of community perceptions and behaviors towards forest environmental services in mitigating human-wildlife conflicts

Nurdin Sulistiyono, Muhdaril Ahda, Ainun Zahira and Shahnaz Dwi Pasha

View article PDF

OPEN ACCESS 012050

Extraction of tannin from guava leaves (*Psidium Guajava L*) by soxhletation method and application as iron corrosion inhibitor in sea water medium

Renita Manurung, Y Simanungkalit and F Amelia

View article PDF

Natural Resource Technology

OPEN ACCESS 012051

Anatomical structures and fiber quality of Jabon (*Neolamarckia cadamba* (Roxb.) Bosser) from agroforestry system

Nelly Anna, Evalina Herawati and Sulivia Pebrianti

View article PDF

OPEN ACCESS 012052

Swelling kinetics of CNC-g-poly (acrylamide-co-graphene oxide) hydrogels at various GO mass

Andika Steven Sitorus, Maya Sarah and Hamidah Harahap

View article PDF

OPEN ACCESS 012053

Batch operations for turbidity reduction in Babura River using carbonized peanut (*Arachis hypogaea* L.) shells with various particle sizes

Bode Haryanto, Farida Hanum, Bungaran Siahaan, Vikram Alexander, Timoty Anugrah Siahaan and Avenia Inkreasinta

View article PDF

OPEN ACCESS 012054

Multistage pectin extraction from lemon (*Citrus limon*) peel: Effect of nitric acid solvent pH and number of stages

Michael, Masytha, Iriany and Taslim

View article PDF

OPEN ACCESS 012055

Enhancement of tensile properties in chitosan/ poly (vinyl alcohol)/ poly(acrylonitrile) polymer electrolyte membranes via ammonium chloride doping

M. Thoriq Al Fath, Michael, Thiodorus Marvin Tjandra, Rosma Natalia Samosir, Gina Cynthia Raphita Hasibuan, Nisaul Fadilah Dalimunthe and Rivaldi Sidabutar

View article PDF

OPEN ACCESS 012056

Effect of reaction temperature on biodiesel production from refined bleached and deodorized palm olein using heterogeneous catalyst from indigofera (*Indigofera zollingeriana*) leaves ash

zollingeriana) leaves ash

Silvia Nova, Taslim, Renita Manurung, Vikram Alexander, Anggara Dwita Burmana and Erlan Rosyadi

View article PDF

OPEN ACCESS 012057

Sustainable forestry: The dual effects of eucalyptus plantations on economy and ecology

Nurhayati Sembiring, Christian Herlim, Abdillah Sani Lubis and Diana Carolina Sitohang

View article PDF

OPEN ACCESS 012058

Integrating natural resources and technological innovation for sustainable growth

Nurhayati Sembiring, Dandi Al-Fiqri Tarigan, S Ingrid Emmanuela, Diana Carolina Sitohang and Muhammad Ghaisan Humam Fasha Azman

View article PDF

OPEN ACCESS 012059

Digital transformation of Lake Toba tourism: Local wisdom-based applications and SEM-analysis on GIS integration in creative economy

Kartini Harahap, Gede Wirata, Nurhayati, Darwin Damanik and Ahmad Faqih Udin

View article PDF

OPEN ACCESS 012060

Landslide susceptibility mapping using a combination of frequency ratio and fuzzy logic methods in The Malino Sub-watershed, Jeneberang Watershed, Indonesia

Andang Suryana Soma, Muh. Dandy Rachmat Ramadhan, Hasnawir and Wahyuni

View article PDF

OPEN ACCESS 012061

Biodiesel synthesis from palm cooking oil utilising sulfuric acid catalyst in a circular microreactor: Optimisation using response surface method

Edy Purwanto and Aloisiyus Yuli Widianto

View article PDF

OPEN ACCESS 012062

Study of the distribution of domestic wastewater from septic tank in silt-type soil

Netti Herlina, Muhammad Faisal, Amir Husin, Edi Yasa Ardiansyah, Emmanuel Gulo and Muhammad Rezki

View article PDF

OPEN ACCESS 012063

Indonesia's policy responses to climate change in sustainable natural resource

management: Jokowi's tenure

Alwi Dahlan Ritonga, Muryanto Amin and Tonny P. Situmorang

View article PDF

OPEN ACCESS 012064

Effect of using three bamboo species as surface layer in hybrid particleboard (HPb)

Nabila, Apri Heri Iswanto, Luthfi Hakim, Widya Fatriasari, Cici Ramadhani, Tito Sucipto, Muhammad Adly Rahandi Lubis, Tomasz Rogoziński and Nam Hum Kim

View article PDF

OPEN ACCESS 012065

Enhancing responsible production and sustainable clean water supply: applying OEE and six big losses analysis in environmental engineering

Yenny Sari, Muhammad Rosiawan, Rifda Aulia and Edy Purwanto

View article PDF

OPEN ACCESS 012066

Effects of board density on the properties of hybrid particleboard from kemenyan wood and belangke bamboo

Apri Heri Iswanto, Bernando Syahputra Lumban Raja, Nabila, Luthfi Hakim, Widya Fatriasari, M. Adly Rahandi Lubis, Tomasz Rogoziński and Nam Hun Kim

View article PDF

OPEN ACCESS 012067

Urban digitalization through clean energy: policies and communication of Medan City government towards "Medan Smart City"

Arief Marizki Purba, Nana Dyki Dirbawanto and Maulana Andinata Dalimunthe

View article PDF

OPEN ACCESS 012068

Diversified utilization of rice field functions for agritourism and education development in Denai Lama Village: Economic sustainability innovation in era 4.0

Emi Triani and Immanuel Cristwo Bangun

View article PDF

OPEN ACCESS 012069

An insight into the development of songket prototype made of yarn derived from oil palm trunk fibers

Arif Nuryawan, Iwan Risnasari, Nella Valensia Sinaga, Tengku Syarfina and Elyse R Stanes

View article PDF

OPEN ACCESS 012070

Processing characteristics and mechanical properties of styrene butadiene rubber filled with varying amounts of silica

I Surya, R Sadanta, L Sukeksi, R Sidabutar and N Hayeemasae

View article PDF

OPEN ACCESS 012071

Synergistic effect of amine-modified palm stearin and carbon black in natural rubber reinforcement applying different cure systems

I Surya, F Az Zahra, M Khatami and H Ismail

View article PDF

OPEN ACCESS 012072

Competitiveness analysis of ecotourism development in Pattunuang nature tourism area of Bantimurung Bulusaraung National Park, Maros Regency

Asrianny, Yovanka Marsanda Paotonan, Nasri and Nuraini

View article PDF

OPEN ACCESS 012073

The properties of green polybag from empty fruit bunch compost and wet decanter solid with chitosan as additive

Ichsan Habibi Hasibuan, Hamidah Harahap and Iriany

View article PDF

Tropical Biodiversity

OPEN ACCESS 012074

Genetic diversity of painted terrapin (*Batagur borneoensis*) using mitochondrial DNA D- Loop Region at Setiu River, Terengganu

Nor Ainsyafikah Madiran, Nur Kaiyisah Suud, Muhammad Syafiq Aiman Mohd Nasir, Norshida Ismail, Muhammad Zaid Nasir, Chik Maslinda Omar and Ahmad Syazni Kamarudin

View article PDF

OPEN ACCESS 012075

Material flow analysis in palm oil plantation

Seri Maulina, Ilham Perkasa Bako, Fitri Rowiyah Rambe, Muhammad Fajar, Nurul Ageng and Ranaf Situmorang

View article PDF

OPEN ACCESS 012076

Estimating the shelf life of instant turmeric rice seasonings with various turmeric

concentrations through Accelerated Shelf Life Testing (ASLT)

Emmanuel Jason and Andreas Romulo

View article PDF

OPEN ACCESS 012077

Haplotype diversity of *Pandanus tectorius* Parkinson from North Sumatran subregions using *trnL-trnF* intergenic spacer

Melfa Aisyah Hutasuhut, Nursahara Pasaribu, Etti Sartina Siregar and Fitmawati

View article PDF

OPEN ACCESS 012078

Macroinvertebrate biodiversity and health status of the upper Wampu River watershed after the eruption of Mount Sinabung

Rusdi Leidonald, Yuli Darnita Br Sinulingga, Eri Yusni, Ahmad Muhtadi, Nur Rohim and Muh Firdaus

View article PDF

OPEN ACCESS 012079

The diversity of macrozoobenthos on Sialang Buah Beach, Serdang Bedagai Regency, North Sumatra Province

Rusdi Leidonald, Cahya Filia Gultom, Amanatul Fadhilah and Ahmad Muhtadi

View article PDF

OPEN ACCESS 012080

Implications of Income Value in the Community Forest Utilization Business Permit Area (IUPHKm) of the Cendana Forest Farmers Group, Enrekang Regency

Adrayanti Sabar, Hasdisyah and Ridwan

View article PDF

OPEN ACCESS 012081

Terrestrial mammal activity in the post-rehabilitation Halaban monitoring area using camera trap data

Mahdiyyah Ardhina, Kaniwa Berliani and Masitta Tanjung

View article PDF

OPEN ACCESS 012082

Structure and composition of plant species and contribution of agrosilvopastura system in Baringeng Village, Libureng District, Bone Regency, Indonesia

Ahmad Rifqi Makkasau, Syamsuddin Millang, Ummi Rosyidah and Muh. Afdal

View article PDF

OPEN ACCESS 012083

Utilization of soil ameliorant and arbuscular mycorrhizal fungi to improve the

growth of *Lagerstroemia speciosa* pers. in subsoil media

Budi Arty, Syamsuddin Millang, Dicky, Nurul Chaerani and Triaty Handayani

View article PDF

OPEN ACCESS 012084

Identification of bee feed plants of *Apis cerana* and honey production in buffer zone of Martelu Purba Natural Reserve

Dwi Endah Widyastuti and Yuni Ayu Permatasari Marbun

View article PDF

OPEN ACCESS 012085

Optimization PGRs concentration using the TIS (Temporary Immersion System) method on Tembesu plants (*Fagraea fragrans* Roxb)

Eunike Christafilia Ruben, Gusmiaty, Iswanto and Nur A'ida

View article PDF

OPEN ACCESS 012086

Estimation and evaluation of potential evapotranspiration calculation models in Medan City

Novita Anggraini, Bagus, Bejo Slamet and Nelly Anna

View article PDF

OPEN ACCESS 012087

Diversity species of leaf litter decomposer fungi *Avicennia marina* at a salinity 11-20 ppt in The downstream of The Deli Belawan River

Yunasfi, Sephia Sembiring, Vinancia Mia S. Sihotang, Sri Rusmayanti Lubis, Gloury Arizona Sitepu, Jhon Ganda Purba and Rikson Silalahi

View article PDF

OPEN ACCESS 012088

Identification of fungi in *Avicennia marina* leaf litter decomposition in the estuary of the Deli River, Belawan District

Yunasfi, Jhon Ganda Purba, Sephia Sembiring, Vinacia Mia S. Sihotang, Gloury Arizona Sitepu and Sri Rusmayanti Lubis

View article PDF

OPEN ACCESS 012089

Plantation of Meranti Bukit (*Shorea platyclados*) with NPK fertilizer treatment at Arboretum Kwala Bekala

Kansih Sri Hartini, Ahmad Baiquni Rangkuti, Bayu Nur Prasetyo and Jefferey Sinaga

View article PDF

OPEN ACCESS 012090

A Review of diospyros species: antibacterial and antifungal compounds, microbiome interactions, and pathogen dynamics

Dwi Sulastri, Hartati, Yeni Khairina, Margaretta Christita, Siti Halimah Larekeng, Iswanto, Rumella Simarmata and Muh Restu

View article PDF

OPEN ACCESS 012091

Diversity of hornbill species (Bucerotidae) at Soraya Research Station, Subulussalam City, Aceh Province

Ma'rifatin Zahrah, Erni Jumilawaty and Nur Halimah

View article PDF

OPEN ACCESS 012092

Araceae diversity in Universitas Sumatera Utara's arboretum

Arida Susilowati, Deni Elfiati, Nurindah Ginting and Ida Mallia Ginting

View article PDF

OPEN ACCESS 012093

Diversity of thalloid liverworts and hornworts in North Sumatra

Etti Sartina Siregar and Ferdinand Susilo

View article PDF

OPEN ACCESS 012094

Food habit of Indian mackerel (*Rastrelliger kanagurta*) landed at the Pantai Labu Fishing Port (TPI) Deli Serdang Regency

Julia Syahrani Hasibuan, Rachel Christy Panggabean, Ipanna Enggar Susetya, Vindy Rilani Manurung and Amanatul Fadhilah

View article PDF

OPEN ACCESS 012095

Reproductive biology of Silver Whiting fish (*Sillago sihama*) landed at the Bagan Deli Fish Landed Site (TPI) Belawan North Sumatra Province

Julia Syahrani Hasibuan, Aisyah Canda Nasution, Vindy Rilani Manurung and Desrita

View article PDF

OPEN ACCESS 012096

The diversity and distribution of Dipterocarpaceae in green space of Universitas Sumatera Utara

Mohnar Maraghiy Harahap, Erni Jumilawaty, Alfan Gunawan Ahmad, Mariah Ulfa, Ida Mallia Ginting, Arel Vincent Waruwu and Pandu Yudha Adi Putra Wirabuana

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