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Biomass to Biofuel: Optimization of Chlorella sp. Harvesting by Using Poly Aluminium Chloride as Coagulant

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ABSTRACT

Chlorella sp. microalgae is promising source of raw material for biodiesel production, owing to the high oil content. To effectively and efficiently harness microalgae, a suitable harvesting process is essential, with coagulation being one of the viable methods. In this study, PAC (Poly Aluminum Chloride) was employed as a coagulant during microalgae harvesting process. Therefore, this study was conducted to optimize the yield of *Chlorella* sp. microalgae by exploring the influence of increased PAC dosage, pH levels, and deposition time. The Box-Bahnken method, incorporated within Response Surface Method (RSM), was employed. The results showed an optimal microalgae yield of 4.95 g, achieved through a combination of an additional dose of 0.75 g/L, a pH level of 10, and a settling time of 10 minutes.

I INTRODUCTION

The world energy consumption is experiencing an increase, attributed to factors such as population growth, mobility, and economic activity. According to the projections by the International Energy Agency-IEA, the global energy demand is anticipated to surge by 45% in 2030, representing an average yearly increase of 1.6%. Approximately 80% of the world energy requirements are expected to be met by fossil fuels [1]. However, the production of fossil fuels, as the primary energy sources, is experiencing a decline owing to their limited and non-renewable nature. Given the concern surrounding energy supply equilibrium, numerous countries have embarked on the search for alternative sources to replace fossil fuels and diminish dependence on non-renewable energy. The utilization of alternative energy sources also yields positive environmental impacts. Energy generated from renewables can be deemed carbon-neutral, thereby making no contribution to carbon emissions in the atmosphere.

One of the most auspicious renewable sources is biomass and Indonesia, being an agriculturally based country, is endowed with a vast abundance of this energy. It can be acquired through the cultivation of crops such as oil palm, coconut, jatropha, or from agricultural residues like rice husks, sugarcane bagasse,

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sawdust, and forestry residues. The Institute for Essential Services Reform (IESR) in 2021 described 4 types of waste with the potential for biomass development, including cocoa husk (0.02 GW), coffee husk (0.03 GW), rice husk (3.08 GW), palm kernel shell (20.3 GW), and acacia wood (7.30 GW) [2]. Microalgae is also one of biomass potential utilized as a biofuel precursor. Generally, microalgae has a high content of lipids up to 40% by mass extracted to obtain algal oil as the feedstock of biodiesel production [3]. Other advantages are easy to cultivate either in the sea or fresh water and can be cultivated in arable land at various operating conditions.

Chlorella sp. is one of microalgae species used as a precursor of biodiesel due to its high oil content about 28 – 32% [4]. The species is categorized in green algae groups, has round shape cells with a diameter range of 2 – 8 µm, reproduces asexually, and its surface is negatively charged. These properties give rise to challenges in the harvesting process, particularly concerning the separation of microalgae biomass from its growing media. The high-speed centrifugation method is considered an effective harvesting technique but the applicability is limited to laboratory-scale operations due to prohibitively high operating costs, rendering it economically unviable for large-scale implementation [5]. As an alternative, the filtration method is commonly employed in large-scale cultivation. However, this method faces constraints when dealing with microalgae of nanoparticle size, such as *Chlorella* or nannochloropsis [6]. To address this issue, microalgae of nanoparticle size can be treated through flocculation and sedimentation. The addition of a coagulant facilitates the formation of floc with microalgae particles, which settle under the influence of gravity, precipitating at the bottom of the sedimentation basin or ponds [7]. Since nanoparticle is negatively charged, cationaic coagulant can form floc by neutralizing the surface. The most widely employed cationic substance used as coagulation is Aluminum ion commonly provided by aluminum sulphate (alum) or Poly Aluminum Chloride (PAC). Aluminum sulfate remains the most commonly used chemical for precipitating particles that float in water media due to its high effectiveness. However, the application of PAC has seen a significant increase owing to its various advantages. These include its ability to generate large floc with a low dosage, a rapid sedimentation rate, low corrosive properties, and the resulting pH change in water during processing is not excessively low [8].

This study aims to determine the optimum condition of *Chlorella* sp. harvesting through the coagulation process by varying PAC dose, time, and initial pH. Optimum process conditions produce very dry biomass of *Chlorella* sp. with acceptable quality. Meanwhile, Response Surface Methodology (RSM) is employed to build a model of experiment design, particularly to determine the variable that significantly influences the response.

II MATERIALS AND METHODS

Cultivation of *Chlorella* sp. was conducted in Nogotirto Algae Park (NAP) by using a transparent plastic container with a capacity of 15 L. The process was carried out by following the standard procedure in NAP regarding media preparation, nutrition, and daily inspection. PAC in technical grade was obtained from a local market, while other chemicals were supplied by Ecolab, Department of Chemical Engineering Universitas Gadjah Mada.

The procedure of harvesting *Chlorella* sp. cultivation was started on day 7 of cultivation. A total of 4 L of *Chlorella* sp. in cultivation media was equalized with various doses of PAC as coagulant. The mixture was stirred up for a setting mixing time and left overnight to form sedimentation. The supernatant in the upper section was withdrawn by using a pump, while the solid phase in the bottom was transferred into filter paper to remove the moisture. The biomass on the filter paper was dried by sunlight for 2 – 3 days to remove the rest of the moisture. Subsequently, the remaining dried biomass was weighed to determine the yield of the harvesting process.

RSM was employed to optimize the operating condition of the harvesting process. A total of three variables were combined, namely dosage of PAC, initial pH of cultivation media, and deposition time. Table 1 summarizes these variables and the variation to be used in RSM analysis. Those minimum and maximum limit values are processed by using Minitab 17 for *Box – Bahnken Design* method to obtain the 15 best randomization samples to be used, as shown in Table 2.

Table 1. Variation of coagulant dose, initial pH, and deposition time

Variable	Symbol	Level		
		-1	0	1
Dose (g/L)	X_1	0,25	0,5	0,75
pH	X_2	8	9	10
Time (minute)	X_3	10	15	20

Table 2. List of harvesting variables samples

Sample ID	Dose (g/L)	pH	Time (minute)	Dry Biomass (g)
1	0,25	8	15	1,35
2	0,75	8	15	1,50
3	0,25	10	15	1,44
4	0,75	10	15	4,52
5	0,25	9	10	1,47
6	0,75	9	10	3,97
7	0,25	9	20	1,21
8	0,75	9	20	3,02
9	0,5	8	10	1,30
10	0,5	10	10	2,40
11	0,5	8	20	1,09
12	0,5	10	20	2,23
13	0,5	9	15	1,87
14	0,5	9	15	1,87
15	0,5	9	15	1,97

Respond variable or dry biomass (Y) was analyzed to produce a linear model, then tested by model fit testing (*Lack of Fit test, concurrent test, and individual test*), Residue assumption test (normality test, and identical test), determination coefficient test (R^2), Plot Contour, and Optimum Variable Point (X_i).

III RESULT & DISCUSSION

Table 2 presents a summary of the dry biomass yield obtained from the experiments. The highest and lowest yield achieved was 4.52 g and 1.09 g in the fourth sample, which used a coagulant dose of 0.75 g/L and 0.50 g/L, a pH level of 10 and 8, and a deposition time of 15 and 20 minutes, respectively. According to the results, it is evident that the optimal dose of PAC for forming floc effectively falls at 0.75 g/L, and this range of coagulant concentration worked well within the pH range of 8 to 10. Interestingly, the deposition time did not have a significant impact on the harvesting process, as longer deposition times did not yield the best results.

To understand the significance of the variable, an analysis of variance (ANOVA) test was carried out, as summarized in Table 3. The results of the Lack of Fit test indicated that the model was appropriate, as the p-value was greater than the significant value ($\alpha = 5\%$). The concurrent test also supported the model acceptability since the p-value for the linear model was less than the significant value ($\alpha = 5\%$). However, individual testing showed that only the coagulant dose variable and pH had a significant effect on the yield of *Chlorella* sp. microalgae, as its respective p-value was less than the significant value ($\alpha = 5\%$). The deposition time did not significantly affect the yield of *Chlorella* sp. microalgae, since its p-value was greater than the significant value ($\alpha = 5\%$).

The mass of the fuel was obtained from the batch system in each testing process through a weighing procedure. The batch process for gasification was expected to make each test more measurable. This approach prevented the addition of fuel which can complicate the prediction of observed variable values during the process.

The next discussion continued with the operational time variable. In this study, the operational time (OT) was used as a reference parameter for observing gasification activities from the point where the fuel was full until everything had been consumed.

Figure 1 shows the Kolmogorv-Smirnov test and the p-value for the normal distribution test. The p-value in the residual normality test is 0.063, meaning the p-value > α (0.05), and the result for H_0 was accepted. Therefore, the residuals had a normal distribution and the assumption that the normality test was fulfilled.

The normality chart in Figure 1 showed the relationship between the actual and predicted values using the residual data. The chart reported that the data was normally distributed since it was spread out evenly around the straight line (red line).

Table 3. Result of analysis of variance

Source	df	Sum of Squares	Mean Square	*P* – *value*
Model	9	14,29	1,58	0,001
Linear *	3	11,00	3,67	0,000
A*	1	7,10	7,02	0,000
B*	1	3,58	3,58	0,001
C**	1	0,32	0,32	0,081
Square	3	1,01	0,34	0,054
A^2	1	0,85	0,85	0,016
B^2	1	0,12	0,12	0,234
C^2	1	0,00	0,00	0,814
Interaction	3	2,27	0,76	0,011
AB	1	2,15	2,15	0,00
AC	1	0,12	0,12	0,239
BC	1	0,00	0,00	0,941
Error	35	0,33	0,07	
Lack of Fit *	3	0,33	0,10	0,030
Pure Error	32	0,00	0,00	
Total	44	14,63		

* Significant

** Not Significant

A = Coagulant dose, B = pH, and C = Deposition time

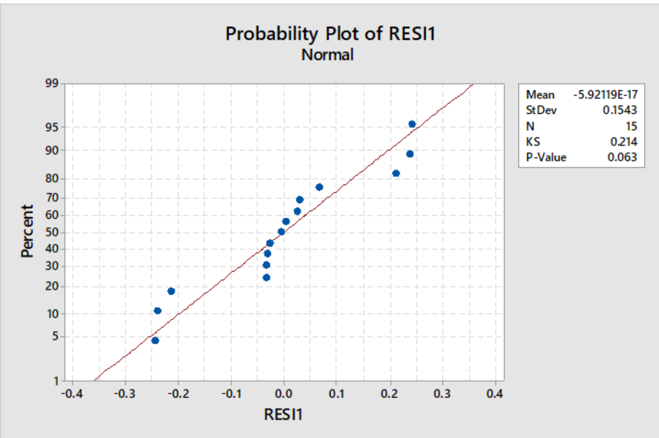

Figure 1. Normal Distribution Chart the Harvesting Process of *Chlorella* sp. Microalgae

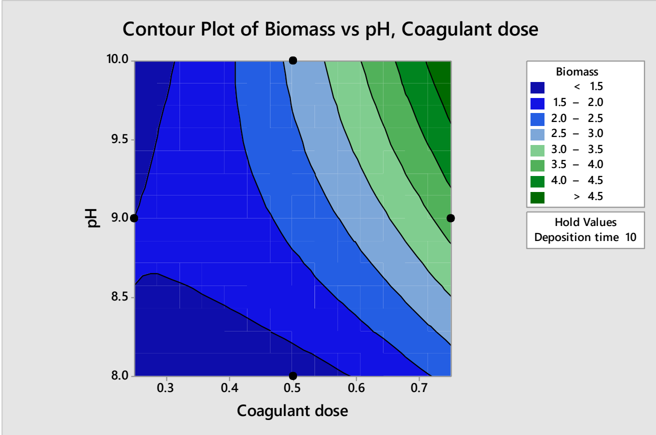

Figure 2. Contour Plot Chart of Coagulant Dose vs pH at Deposition Time = 10

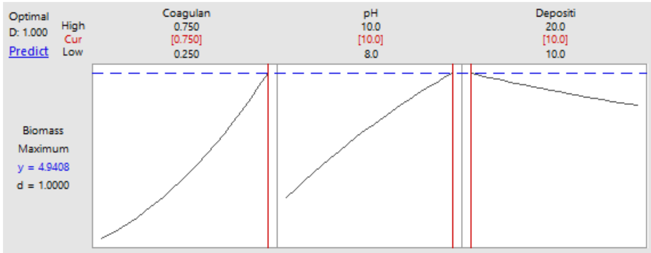

Figure 3. Optimization Plot Chart the Harvesting Process of *Chlorella* sp. Microalgae

The contour plot in Figure 2 showed the response (microalgae yield) to the combination of three variables using a 2D contour plot. The lines on the plot reported that these three variables affected the yield of *Chlorella* sp. microalgae. The optimal point, as shown in the plot, was achieved with a coagulant dose of 0.75 g/L, a pH of 10, and a deposition time of 10 minutes, resulting in a dry biomass weight of 4.95 g/4 L, as shown in Figure 3.

According to Table 3, the addition of a coagulant such as PAC and the pH of the culture significantly influenced the yield of *Chlorella* sp. microalgae. This was because the addition of a cationic coagulant neutralized the negative charge in the culture yield and caused floc formation. The higher the dose of PAC, the more cations were released into the culture media [9]. According to its nature, PAC functioned in a wide pH range of 6 to 10 and did not affect the coagulation process [10]. Rachmawati et al. (2009) also found that an increase in coagulant dose enhanced the deposition process and the pulling force, thereby widening the pH range of use [11].

The deposition time did not significantly affect the yield of microalgae, as supported by Karamah and Lubis (2014). After the floc size had formed and precipitation occurred, additional coagulation time was not necessary, since the formed floc did not continue to grow[12].

IV CONCLUSION

In conclusion, the harvesting process of *Chlorella* sp. cultivation was successfully optimized using PAC as a coagulant. According to the Box-Bahnken method of RSM, the addition of coagulant dose and pH had a significant effect on the yield of *Chlorella* sp. microalgae. The optimum yield was 4.95 g with a combination of 0.75 g/L coagulant, a pH of 10, and a deposition time of 10 minutes.

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