# **RESEARCH ARTICLE**



Microwave-Assisted Hydrolysis of Robusta Coffee Parchment as a

# Reducing Sugar Feedstock

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Abstract. The hydrolysis process is a process of breaking down substances by reacting using water, the aim is to break down the substance. This research focuses on the hydrolysis process to determine the reducing sugar content of coffee parchment. The method used is microwaveassisted hydrolysis. This method can increase lignin release more effectively than conventional methods in the process of cellulose and hemicellulose hydrolysis. The most commonly used hydrolysis to hydrolyze cellulose is acid hydrolysis. This research uses coffee parchment raw material (horn skin) which contains cellulose and hemicellulose with HCl solvent. Hydrolysis with acid concentration is  $(1, 2, \text{ and } 3\%)$ , microwave power  $(150, 300, \text{ and } 450 \text{ W})$ , and time (20, 25, and 30 minutes). In this study, the optimal reducing sugar yield was 8,054 g/mL under the operating conditions of 25 minutes, 3% HCl concentration, and 450 W microwave power.

**Keywords:** *Hydrolysis, coffee parchment, microwave-assisted hydrolysis, and reducing sugar.*

# **1. Introduction**

Over time the need for fuel has increased, requiring alternative fuels. Fossil fuel reserves are dwindling, even though people's needs are increasing. In addition, as fuel oil becomes increasingly expensive, there is a growing need for alternative fuel sources such as biomass. Currently, Indonesia's petroleum reserves are only 1% of the world's oil reserves, therefore there is a need for other alternatives to replace the use of kerosene and LPG gas which are currently still widely used by the community [1]. Second-generation bioethanol with cellulose raw materials derived from various biomass materials is one form of alternative energy. The potential of cellulose waste from wood and non-wood materials is the most abundant natural resource, one of which is coffee parchment [2].

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Based on data from the Central Statistics Agency (BPS), coffee availability in Indonesia in 2020 reached 99.33% when combined with state-owned and private plantations. This shows the amount of coffee production that is exported both domestically and abroad. The availability of 45% coffee parchment in East Java, especially in the area around Bondowoso [3]. Coffee parchment as raw material can produce bioethanol with 38.68% content after going through hydrolysis and fermentation processes. The advantage of cellulose conversion into various products can also reduce environmental pollution problems due to the accumulation of agricultural biomass waste that is not optimally utilized [4]. Bioethanol is an organic fuel derived from plant materials through the natural process. However, there are various methods for producing bioethanol, such as the fermentation process involving microorganisms [5]. Bioethanol includes wood waste, agricultural waste, plantations, forest products, and organic components from industry and households [6].

As one of the alternative fuel sources processed from plants, bioethanol has the advantage of being able to reduce  $CO<sub>2</sub>$  emissions [7]. According to Wusnah et al, 2019 [8], sources of bioethanol are starch-containing plants (such as cassava, oil palm, tengkawang, coconut, kapok, jatropha, rambutan, soursop, malapari, and nyamplung), sugary (such as molasses, palm sap, sugarcane sap, and sweet sorghum sap) and cellulose fibers (such as sorghum stems, banana stems, straw, wood, and bagasse) [9]. Bioethanol has advantages compared to fuel, including having a higher oxygen content of about (35%) so that it burns more completely, a higher octane value (118), and is more environmentally friendly because it has a lower CO gas emission content of 19-25% [10]. One example of bioethanol raw material is coffee parchment, the availability of coffee parchment is quite large, in coffee processing will produce 65% coffee beans and 35% coffee parchment [11]. The structure of the coffee fruit can be seen in Figure 1 and the constituent content of robusta coffee can be seen in Table 1.



**Figure 1.** Structure of Coffee Fruit

Parchment has a fairly high cellulose content, making it possible to produce more glucose. According to Wardana et al, 2019 [4] the cellulose and hemicellulose content in Robusta coffee parchment after dry method treatment was found to be 27.26% cellulose and 11.65% hemicellulose, where the highest levels in the Bondowoso region were greater than other regions. The advantage of using Robusta coffee parchment as a raw material is that the fruit is widely grown around the Bondowoso district and has a high enough cellulose content so it can produce high glucose.

<b>Outer skin</b>	Fruit pulp	Parchment	<b>Epidermis</b>	<b>Seed</b>
Crude protein	Pectin	Crude protein	Chlorogenic acid	Crude fiber
(91.7%)	$(38.70\%)$	$(2.20\%)$	$(7.21\%)$	(27.2%)
Fat	Total sugar	Crude fiber	Fat	fat
(2%)	$(45.80\%)$	$(60.24\%)$	(10.19%)	$(10.6 - 12.6\%)$
Fiber	water	Cellulose		Crude protein
(27.65%)	(15.15%)	(63%)		$(3-13.5%)$
Reducing sugar		Hemicellulose		Reducing sugar
$(12.4\%)$		$(7.58\%)$		$(6-10\%)$
Non-reducing sugar		Ash		Non-reducing sugar
$(2.02\%)$		$(3.30\%)$		$(0.32 - 1.08\%)$
Tannin				Carbohydrates
(7.47%)				$(4.47\%)$
Total pectin				Ash
$(6.52\%)$				$(3.38\%)$
Ash				Caffeine
$(3.36\%)$				(9%)
N free				Chlorogenic acid
(57.85%)				$(6-10\%)$

**Table 1.** Ingredients of Robusta Coffee

Based on Table 1, coffee skin contains 63% cellulose and 7.58% hemicellulose. Coffee parchment is an abundant agro-industrial cellulosic and lignocellulosic material that can be used to produce reducing sugars. The composition of coffee parchment consists of cellulose, hemicellulose, and lignin. However, the high lignin content requires pretreatment [12]. With its considerable cellulose content, the utilization of coffee skin as a raw material for reducing sugar with the application of bioethanol can be an opportunity for non-conventional fuels.

Bioethanol can be made from various agricultural materials, including materials containing sugar derivatives (saccharin), starch-containing materials, and cellulose-containing materials such as wood, and some other agricultural waste [10]. Materials containing saccharin can be directly fermented, but materials containing starch and cellulose must first be hydrolyzed into simple components, although they can be fermented directly using enzymes, currently the fermentation industry still utilizes microorganisms, because this method is much easier and cheaper, microbes that are widely used in the fermentation process are yeasts, molds, and bacteria [13]. The hydrolysis process is a process of breaking down substances by reacting using water, the aim is to break down these substances [12]. The purpose of this study is to optimize the hydrolysis process of coffee parchment using a microwave to determine the highest level of reducing sugar. Box–Behnken design (BBD) and response surface methodology (RSM) are employed to obtain statistical models for glucose stability optimization and identification of their stability regions. Factors including the HCL concentrations, hydrolysis time, and power microwave are subjected to sensitivity analyses, highlighting their ascendancy and interactional effect on the formulation stability.

The novelty carried out in this study is to vary the time, acid concentration, and power in the hydrolysis process of parchment, it is expected to obtain more optimal reducing sugar to produce the best bioethanol. According to Maryanti et al, 2019 [14], the most used hydrolysis to hydrolyze cellulose is acid hydrolysis. Some acids commonly used for acid hydrolysis include sulfuric acid ( $H_2SO_4$ ), perchloric acid ( $HCIO_4$ ), and hydrochloric acid ( $HCI$ ). The use of concentrated acid in the cellulose hydrolysis process is carried out at a lower temperature than dilute acid. The concentration of concentrated acid used is 1-3%. The reaction temperature is 100˚C and requires a reaction time between 20-40 minutes [15].

Microwaves are electromagnetic waves that have frequencies from 0.3 to 300 GHz. Lignin can be broken into smaller particles and detached from cellulose at 200ºC, pretreatment using microwave can be achieved within 60 minutes [16]. The use of microwaves enhances the release of protein bodies from starch more effectively than traditional methods, not only in the pretreatment process but also in the cellulose and hemicellulose hydrolysis process. In the hydrolysis process of these two materials in an acidic solution using the microwave method, starch is directly converted into simple sugars in a relatively short time. Compared with traditional heating, the reaction rate of starch hydrolysis to glucose is increased by 50-100 times [5]. The advantage of using this acid is that it contains sugar conversion up to 90% conversion [17]. The initial moisture content of robusta coffee itself is around 48.7% and according to SNI, the maximum moisture content of dried coffee bean skin is 12.5% [18]. The results of glucose hydrolysis of robusta coffee obtained 6.73% and the results of glucose fermentation of robusta coffee skin for 7 days obtained a bioethanol content of 60%. The conversion process of glucose to produce bioethanol occurs maximally [19]. Previous research data can be seen in Table 2.

Some of the research results in the table obtained the highest reducing sugar yield of 42 g/mL using the acid hydrolysis method. The novelty of this research is that it uses the parameters of hydrolysis time, HCl concentration, and microwave power so that it influences the results of reducing sugar levels.

Raw <b>Material</b>	<b>Hydrolysis</b> <b>Method</b>	<b>Operating Conditions and Result</b>	Reference
Robusta coffee parchment	Acid hydrolysis	The results of reducing water content obtained optimal results for 2 hours with water content lost by 12.056%, with a ratio of 8% hydrolysis starter obtained the highest reducing sugar of 673.765 mg/100 mL.	[10]
Water hyacinth	Acid hydrolysis	The best water hyacinth hydrolysis process conditions obtained were the use of 1 NH <sub>2</sub> SO <sub>4</sub> catalyst and 600 W microwave power with a final reducing sugar content of 486 mg/L.	[16]
Rice straw	Acid hydrolysis using hydrolysis flask	Under optimal conditions, the highest reducing sugar yield from hydrolysis for 25 hours with 40% enzyme concentration was obtained at 8.75 mg.	$[20]$
Coffee skin Arabica	Acid hydrolysis	The highest reducing sugar content was obtained at $42.6$ g/mL. Based on the 42% cellulose yield, the highest reducing sugar	[13]
coffee parchment	Acid hydrolysis	was obtained from the hydrolysis with 10% H <sub>2</sub> SO <sub>4</sub> at 100°C by $8%$ .	$[21]$
Corn cob	Enzymatic hydrolysis with autoclave heating	Hydrolysis with cellulase enzyme, in alkaline treatment and repetition of 3 times obtained reducing sugar content of 9.96%.	$[22]$
Sorghum dregs	Microwave aqueous acid hydrolysis	By varying the concentration for 30 and 40 minutes, it was found that the reducing sugar content increased with increasing acid concentration at 30 minutes of hydrolysis time, namely 2.0-8.5 mg/L and experienced a significant increase when extending the hydrolysis time to 40 minutes, namely 19.1-42.7 mg/L. The largest reducing sugar concentration for ethanol production using 150°C temperature with $2\%$ acid concentration at 40 minutes hydrolysis time is 34.3 mg/L.	$\lceil 5 \rceil$
Sorghum Dregs	Acid hydrolysis using autoclave and microwave	The highest glucose level from the hydrolysis process using autoclaving obtained the highest reducing sugar of 30.86 g/L, with hydrolysis using microwave obtained reducing sugar of 44.97 g/L.	$[23]$
Cassava peel	Acid hydrolysis	From the hydrolysis process, the reducing sugar content was 9.9%.	$[24]$
Pineapple peel	Acid hydrolysis with stirring rate	From the results of this study, the optimal conditions of hydrolysis obtained the highest glucose occurred from the addition of HCl at 2 M and 300 RPM at 12.6%.	$[9]$

**Table 2.** Previous Research Data of Bioethanol Reducing Sugar Hydrolysis



#### **2. Materials and Methods**

#### 2.1 Materials

The materials used in this study were robusta coffee parchment obtained from Sumber Wringin Village, Bondowoso District, East Java, distilled water, HCl 32%, NaOH 48%, anhydrous glucose, KNa tartrate, and 3.5-Dinitrosalicylic Acid.

#### 2.2 Equipment

Blender (Miyako), 100 mesh sieve (RSL stainless steel 304), microwave (Samsung MS23K3515AS/SE), water bath (Prio WB-2-6), UV-Vis spectrophotometer (Tungsten 6mm).

#### 2.3 Method

In this study, it was determined that: The control variable is the mass of coffee parchment of 100 grams with a size of 100 mesh [11]. The dependent variable is the reduced sugar content in the hydrolysis process. The independent variables used are hydrolysis time (20, 25, and 30 minutes) [15], acid concentration (1, 2, and 3%) [5], and Microwave power (150, 300, and 450 W) [26]. The analytical method used in this research is Box-Behnken Design (BBD) and response surface methodology (RSM) to obtain a statistical model for optimizing glucose stability and identifying areas of stability.

#### 2.3.1 Raw Material Preparation

Robusta coffee parchment was cleaned first and then dried using an oven at 100˚C for 15 minutes and obtained a sample with a moisture content of 9.998%. Then, the coffee skin is pulverized using a blender so that the results are obtained in powder form. Furthermore, the coffee skin powder is sieved using a sieve with a size of 100 mesh [11].

#### 2.3.2 Hydrolysis of Parchment

The coffee powder material weighed as much as 10 g with HCl catalyst concentration (1, 2, and 3%) as much as 100 mL then put into the flask to be hydrolyzed using microwave heating for (20, 25, and 30 minutes) with microwave power (150, 300, and 450 W) and then allowed to stand until the temperature of the hydrolysis results dropped to room temperature (25). This study carried out data analysis using Design Expert 11 Version Software with Response Surface Methodology (RSM) BBD (Box Behnken Design) model 17 times shown in Table 3, then analyzed the reducing sugar content by DNS method.

	<b>Time</b>	Concentration	Power
Run	(minutes)	(g/mL)	(W)
	20	$\overline{2}$	450
	30	2	450
3	25		300
4	30		300
5	20		300
6	25		300
	25		450
8	25		300
9	20	2	150
10	25		150
11	25		450
12	20		300
13	25		150
14	30	2	150
15	25	2	300
16	30		300
17	25		300

**Table 3**. Variation of Box-Behnken Design (BBD) Acid Hydrolysis with 3 Variables Data

# 2.3.3 Reducing Sugar Analysis

Analysis of reducing sugars based on preliminary research on analyzing glucose levels using a UV-Vis Spectrophotometer using DNS reagents conducted by Kolo [5], Wardani [27], and Santi [28].

#### 2.3.4 Preparation of DNSA Reagent

A total of 1 g of 3.5-Dinitrosalisilic was added to 20 mL of NaOH and homogenized. Separately dissolved KNa Tartrate (KNa  $C_4H_4O_6.4H_2O$ ) as much as 30 g using distilled water and homogenized both solutions. Then put into a 100 mL volumetric flask, added distilled water until it reached the limit, homogenized, and transferred into a bottle [23].

# 2.3.5 Glucose Standard Curve Preparation

Standard glucose solutions were made with concentrations of 200, 400, 600, 800, and 1000 ppm, respectively. The preparation begins with making a glucose mother solution by dissolving 100 mg of anhydrous glucose and adding 100 mL of distilled water. Each standard glucose solution was taken 1 mL and placed in each test tube. Then added 1 mL of 3.5- Dinitrosalicylic Acid reagent and 1 mL of distilled water, then homogenized and heated in a water bath with boiling water for 5 minutes. Next, the solution was cooled to room temperature and added distilled water to a volume of 5 mL, and homogenized again. Absorbance was measured using a UV-Vis Spectrophotometer with a wavelength of 540 nm. Absorbance is the ratio of the intensity of the light fired to the intensity of the absorbed light, the absorbance value will increase with the results obtained. Absorbance measurements were taken at each concentration of glucose solution and then a standard curve plot was made with glucose content (mg) abscissa  $(x)$  and absorbance as ordinate  $(y)$  [29].

# 2.3.6 Determination of Reducing Sugar

1 mL of sample was taken to the test tube, 0.5 mL of DNSA reagent was added, 2 mL of distilled water was homogenized, and the sample solution was heated in a water bath for 5 minutes. Then, the solution was cooled to room temperature, and added distilled water until the final volume of 5 mL, then tested using a UV-Vis spectrophotometer with a wavelength of 540 nm.

## **3. Result and Discussion**

3.1 Analysis of Reducing Sugar Content



**Figure 2.** Glucose Standard of Solution Curve

The determination of reducing sugar content in this study was carried out first by determining the glucose standard curve to determine the linear regression equation. This curve was used to determine the concentration of reducing sugar in the samples. The glucose standard curve was obtained by measuring the absorbance of glucose standard solution with various concentrations of 200, 400, 600, 800, and 1000 ppm. Figure 2 shows that the curve is obtained from the linear regression equation at  $y = 0.0011x - 0.001$ , with an  $R^2$  value of 0.9982. The results of reducing sugar can be seen in Table 4.

Run	$\mathbf{A}$ <b>Time</b> (minutes)	$\bf{B}$ Concentration $(\%)$	$\mathbf C$ Power (W)	<b>Reduced Sugar</b> Content (g/mL)
$\mathbf{1}$	20	$\overline{2}$	450	4.554
$\overline{2}$	30	$\overline{2}$	450	7.059
3	25	$\overline{2}$	300	6.436
$\overline{4}$	30	$\mathbf{1}$	300	4.190
5	20	$\mathbf{1}$	300	4.050
6	25	$\overline{2}$	300	6.927
7	25	$\overline{3}$	450	8.054
8	25	$\overline{2}$	300	7.286
9	20	$\overline{2}$	150	3.950
$10\,$	25	$\mathbf{1}$	150	3.731
11	25	$\mathbf{1}$	450	3.977
12	20	$\overline{3}$	300	5.095
13	25	$\overline{3}$	150	4.840
14	30	$\overline{2}$	150	5.004
15	25	$\overline{2}$	300	6.150
16	30	$\overline{3}$	300	7.254
17	25	$\overline{2}$	300	7.163

**Table 4.** Results of reducing sugar content of robusta parchment

Based on Table 4, measured using a UV-Vis spectrophotometer, the highest reducing sugar content was 8.054 g/mL in the operating conditions of 25 minutes, 3% HCl concentration, and 450 W microwave power. Research with the microwave hydrolysis method conducted by Putera et al, 2019 [10], had lower results than this study, which amounted to 6.737 g/mL with an HCl concentration of 20% within 2 hours with an absorbance range of 3.22%.

# 3.2 *Analysis of Variance* (ANOVA)

The ANOVA result for this study can be seen in Table 5.

<b>Source</b>	Sum of <b>Squares</b>	df	Mean <b>Square</b>	<b>F-value</b>	p-value	
<b>Model</b>	33.08	9	3.68	18.88	0.0004	significant
A-Time	4.29		4.29	22.04	0.0022	
B-Concentration	10.80		10.80	55.48	0.0001	
C-Power	4.68		4.68	24.04	0.0017	
AB	1.02		1.02	5.23	0.0560	
AC	0.5256		0.5256	2.70	0.1443	
BC	2.20		2.20	11.31	0.0120	

**Table 5**. Analysis of Variance (ANOVA) Results

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Analysis of variance (ANOVA) is to determine whether these factors have a significant effect on variables with a form of statistical hypothesis testing where conclusions are made based on data or inferential statistical groups [30]. Based on Table 5, the variables of time (A), concentration (B), and power (C) used in this study have a significant effect on reducing sugar. This can be seen from the p-value (probability value) for time 0.0022, HCl concentration 0.0001, and power 0.0017 showing that the results of the analysis are smaller than the probability value which is  $\leq 0.05$  (5%).

Based on Table 5, the lack of fit value at the p-value is 0.6479, which represents that the ANOVA model produced passes the principle of fit. The lack of fit value is insignificant, lack of fit can be used if the value is  $\geq 0.05$ . Lack of fit is a deviation or inaccuracy of independent variables against a model [31]. Fit statistics for this study can be seen in Table 6.

<b>Tapic v. I</b> wouldn't					
$\mathbf{R}^2$	Adjusted $\mathbb{R}^2$	Predicted $\mathbb{R}^2$	<b>Adeq Precision</b>		
0.9604	0.9096	0.7608	12.0036		

**Table 6**. *Fit Statistic*

The values of  $\mathbb{R}^2$ , adj- $\mathbb{R}^2$  and pred- $\mathbb{R}^2$  are important to note. These values provide an expectation of the accuracy of an independent variable in the model. A good model has a minimum  $\mathbb{R}^2$  value of 0.8 or 80%. The value of  $\mathbb{R}^2$  is closer to 1, the better the resulting model [33]. Based on Table 6, the Fit Statistic data was obtained in this study. It is known that the value of  $\mathbb{R}^2$  is 0.9604, this indicates that hydrolysis time, HCl concentration, and microwave power have an influence of 0.9604 or 96.04% on reducing sugar. The adjusted  $\mathbb{R}^2$  value (adjusted) is 0.9096, then the predicted  $\mathbb{R}^2$  value is 0.7608 so that it can be accepted as appropriate because it has a difference  $\leq 0.2$  [32]



**Figure 3.** Comparison Chart of Model Data (Predicted) with Experiment (Actual)

Figure 3 shows the fit of the model data to the experimental data can be depicted on the pority plot graph. The straight line on the graph is the prediction data, while the actual data for each experimental data is depicted as a point on the graph. It can be seen from the resulting graph that there are some actual data obtained close to the predicted data.

The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. The mathematical equation between the independent variable and the dependent variable to determine the reducing sugar content is shown in equation (2). The positive value in the equation is the response of reducing sugar content will increase directly proportional to the heating time with HCl concentration, the interaction between heating time and microwave power, the interaction between concentration and microwave power, and vice versa. The response of reducing sugar content will decrease as the HCl concentration decreases, this is indicated by the negative coefficient of the equation.

$$
y = 1.4537A + 0.4269B + 0.005C + 0.1009AB + 0.00048AC + 0.00049BC - 0.3308A^2 - 0.8179B^2 - 0.000037C^2
$$
 (2)

where,

 $Y =$  Reducing sugar content  $(g/mL)$ 

 $A =$  Heating time (minutes)

 $B = HC1$  concentration  $(\%)$ 

 $C =$ Microwave power (W)

- 3.3 Effect of Hydrolysis Variables on Reducing Sugar Content
- 3.3.1 The effect of the relationship between time and HCl concentration on reducing sugar content response.



**Figure 4**. Effect of Hydrolysis Time (A) on HCl Concentration (B)

Figure 4 shows the reduced sugar content as affected by time (A) and HCl concentration (B). The reduced sugar content influenced by hydrolysis time is shown in Table 4. At a hydrolysis time of 20 minutes with 1% HCl concentration and 300 W of power, the reducing sugar content was 4.05 g/mL. If the time is increased to 25 minutes with 2% HCl concentration and 300 W of power, the result of reducing sugar is 7.16364 g/mL. the longer the hydrolysis time, the reduced sugar content. However, if it exceeds the optimum time, the reducing sugar content will also decrease due to inhibitors usually formed by hydroxymethylfurfural (HMF) compounds, too much acid concentration or too high hydrolysis temperature can increase the amount of inhibitors formed during the process so that it can inhibit glucose formation [10]. This is following with the research of Rifa'I et al, 2019 [6], so the time and HCl concentration variation has a significant effect on the response of reducing sugar content



3.3.2 The effect of the relationship between time and microwave power on reducing sugar content response

**Figure 5.** Effect of Hydrolysis Time (A) and Microwave Power (C)

Figure 5 shows the graph of the relationship between time (A) and power (C) to the response of reducing sugar content, optimal condition was obtained in the  $7<sup>th</sup>$  experiment. It is known that at a time of 20 minutes and a concentration of 2% HCl with 150 W of power, the result of reducing sugar is 3.95 g/mL. when increased to 25 minutes and a concentration of 2% HCl with 300 W of power, the result of reducing sugar is 7.28636 g/mL. In this case, as the microwave power increases, the reduced sugar content will also increase. So, it can be said that if the time variable is increased with the experiment, it also accelerates the hydrolysis time in accordance with previous research conducted by Amini et al, 2022 [3].



3.3.3 The effect of the relationship between HCl concentration and microwave power on reducing sugar content response.

**Figure 6.** Effect of HCl Concentration (B) and Microwave Power (C)

Figure 6 shows the graph of the effect of the relationship between HCl concentration (B) and microwave power (B) to reducing sugar, obtained optimal results that occurred in the  $7<sup>th</sup>$  experiment with a reducing sugar yield of 8.05455 g/mL. At a time of 25 minutes and a concentration of 1% HCl with a microwave power of 150 W, the reducing sugar was obtained at 3.73182 g/mL. If the HCl concentration is enlarged in the 25-minute experiment and 2% HCl concentration with 300 W of microwave power, the result of reducing sugar is 7.28636 g/mL, from these results it can be concluded that the addition of HCl has a significant effect on the increase in reducing sugar. This is comparable to the statement of Ahmad et al, 2020 [34]. It is known that the variation in HCl concentration has a linear effect along with the increase in  $H<sup>+</sup>$ ions on the glucose content produced. However, if the addition of acid concentration is increased beyond the optimum point, it will undergo decomposition into HMF compounds [33].

Based on Table 7, the optimal result on the response of one experiment on reducing sugar content is 8.021 g/mL when the hydrolysis time is 29.4, HCl concentration is 2.6%, microwave power is 370.8 W and also the desirability value reaches 1.000. The suitability of the model to the optimization value is obtained if the desirability value is close to one [34].

<b>Hydrolysis</b> Time	<b>HCL</b> Concentration	<b>Microwave</b> <b>Power</b>	<b>Reduced</b> Sugar Contet	<b>Desirability</b>
29.4	2.6	370.8	8.021	1.000

**Table 7.** Maximum optimization of reducing sugar content design expert

# 3.4 Comparison of Reducing Sugar with Previous Research

The following Table 8 provides data on the results of previous studies as a reference for comparison with this study.

No.	<b>Material</b>	<b>Methods</b>	<b>Reduced Sugar</b> <b>Content Result</b>	<b>Reference</b>
1.	Robusta coffee	HCl, Agitation	$6.73$ g/mL	[10]
2.	Arabica coffee	$H2SO4$ , Hot Plate	$4.86$ g/mL	$\lceil 21 \rceil$
3.	Robusta coffee	HCl, Water <b>Bath Shaker</b>	$20.85$ g/mL	[4]
4.	Robusta coffee	Kapang Pestalotiopsis sp. VM 9, Inokulasi	$3.92$ g/mL	$\left[35\right]$
5.	Kopi Robusta	HCl, Microwave- Assisted hydrolysis	$8.05$ g/mL	This study

**Table 8.** Comparison of Reducing Sugar Levels with Previous Studies

# **4. Conclusions**

Based on the results of the study, it can be concluded that the highest reducing sugar content of 8.054 g/mL is in the 7th run with 3% HCl concentration, 25 minutes hydrolysis time, and 450 W microwave power. Determination analysis using Analysis of variance (ANOVA) obtained the results that a significant effect occurred on all variables, namely hydrolysis time, HCl concentration, and microwave power with an R-value of 0.9096 where the value is close to 1, the better the experimental results. If the hydrolysis process passes the optimal conditions, the increase in reducing sugar will decrease because other compounds are formed, namely HMF.

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## **REFERENCES**

- [1] V. Serevina, R. D. Pambudi, D. A. Nugroho, U. N. Jakarta, and J. Timur, "Pelatihan pemanfaatan limbah gergaji dan cangkang telur ayam untuk membuka usaha briket biomassa," vol. 1, no. 1, pp. 1–5, 2021.
- [2] A.-A. Nainia, Nurwahdaha, R. Y. Lestari, and Sunardi, "Praperlakuan secara hidrotermal limbah lignoselulosa untuk produksi bioetanol generasi kedua," *J. Ris. Ind. Has. Hutan Vol. 10 (2), 2018, 93 …*, vol. 10, no. 2, pp. 93–102, 2018, [Online]. Available: http://eprints.ulm.ac.id/9372/1/Publikasi 13.pdf
- [3] H. W. Amini *et al.*, "Ekstraksi Limbah Kulit Kopi Robusta Dari Desa Tanah Wulan Kecamatan Maesan Kabupaten Bondowoso Dengan Etil Asetat Serta Analisa Aktivitas Antioksidannya," *e - Pros. Kolok. Has. Penelit. dan Pengabdi. Kpd. Masy.*, pp. 87–92, 2022.
- [4] D. I. Wardhana, E. Ruriani, and A. Nafi, "Karakteristik Kulit Kopi Robusta Hasil Samping Pengolahan Metode Kering Dari Perkebunan Kopi Rakyat Di Jawa Timur," *Agritrop J. Ilmu-Ilmu Pertan. (Journal Agric. Sci.*, vol. 17, no. 2, p. 214, 2019, doi: 10.32528/agritrop.v17i2.2569.
- [5] S. M. D. Kolo and Y. Sine, "Produksi Bioetanol dari Ampas Sorgum Lahan Kering dengan Perlakuan Awal Microwave Irradiasi," vol. 2, no. 2622, pp. 39–40, 2019.
- [6] A. F. Rifa'i, W. A. Pamungkas, R. B. Setyawati, C. P. Setiawan, and J. Waluyo, "Kajian Teknoekonomi Bioetanol Berbahan Molasses Sebagai Alternatif Substitusi BBM," *Equilib. J. Chem. Eng.*, vol. 6, no. 1, p. 61, 2022, [Online]. Available: https://jurnal.uns.ac.id/equilibrium/article/view/63158
- [7] M. Djana, "Pengaruh Massa Ragi Dan Lama Fermentasi Terhadap Pembuatan Etanol Dari Enceng Gondok," *Integr. J. Ilm. Tek. Ind.*, vol. 1, no. 2, pp. 36–44, 2018, [Online]. Available: https://jurnal.um-palembang.ac.id/integrasi/article/download/1000/874
- [8] W. Wusnah, S. Bahri, and D. Hartono, "Proses Pembuatan Bioetanol dari Kulit Pisang Kepok (Musa acuminata B.C) secara Fermentasi," *J. Teknol. Kim. Unimal*, vol. 8, no. 1, p. 48, 2019, doi: 10.29103/jtku.v8i1.1915.
- [9] L. Nulhakim *et al.*, "Pembuatan Bioetanol dari Kulit Nanas Oleh Saccharomyces Cerevisiae Terimobilisasi dalam Butiran Alginat," *Semin. Nas. AVoER XI*, vol. 24, no. 1, pp. 444–448, 2019.
- [10] M. Akbar, H. Putra, and H. Purnama, "Pengaruh Waktu Pengeringan dan Rasio Bahan Baku/Starter Zymomonas Mobilis Pada Pembuatan Bioethanol dari Limbah Kulit Kopi Robusta," *Simp. Nas. RAPI XVIIII – 2019 FT UMS*, no. 1963, pp. 420–425, 2019.
- [11] . S. and M. Syabriana, "Produksi Bioetanol Dari Limbah Kulit Kopi Menggunakan Enzim Zymomonas Mobilis Dan Saccharomyces Cereviseae," *J. Serambi Eng.*, vol. 3, no. 1, pp. 271–278, 2018, doi: 10.32672/jse.v3i1.356.
- [12] D. F. Nury, M. Z. Luthfi, and Y. Variyana, "Pengaruh Pretreatment Alkali Hidroksida Terhadap Produksi Gula Reduksi dari Limbah Kulit Kopi," vol. 1, no. 1, pp. 1–6, 2023.
- [13] R. F. Azzahra and Meilianti, "Produksi Bioetanol Berbahan Dasar Limbah Kulit Kopi

Sebagai Bahan Bakar Alternatif," *J. Kinet.*, vol. 12, no. 02, pp. 58–63, 2021.

- [14] Y. Maryanty, F. L. W. Saputra, and R. Prasetyo, "Pembuatan Asam Laktat dari Selulosa oleh Bakteri Lactobacillus delbrueckii dengan Selulase dari Bakteri Bacillus subtilis dan Bacillus circulans," *J. Tek. Kim. dan Lingkung.*, vol. 4, no. 2, p. 153, 2020, doi: 10.33795/jtkl.v4i2.179.
- [15] S. M. D. Kolo, "The Effect of Hydrolysis Time Using Microwave on Bioethanol Production from Sorghum Waste (Sorghum Bicolor L.)," *J. Sains dan Terap. Kim.*, vol. 16, no. 1, p. 28, 2022, doi: 10.20527/jstk.v16i1.11404.
- [16] M. Sarah, I. Pratiwi, and I. M. Hasibuan, "Hidrolisis Eceng Gondok (Eichhornia Crassipes) Menjadi Glukosa Menggunakan Rotating Microwave Reactor," *J. Tek. Kim. USU*, vol. 11, no. 1, pp. 49–55, 2022, doi: 10.32734/jtk.v11i1.6760.
- [17] N. Nurkholis, N. R. Afifah, and S. Nealma, "Sintesis Bioetanol Dari Buah Berenuk (Crescentia cujete L.) Dengan Metode Hidrolisis Asam Dan Fermentasi Alkoholik," *J. Teknol.*, vol. 6, no. 2, pp. 99–106, 2019, doi: 10.31479/jtek.v6i2.26.
- [18] A. Teniro and Z. Zainudin, "Optimalisasi Pengolahan Biji Kopi Dalam Upaya Peningkatan Pendapatan Petani," *J. Pengabdi. Pada Masy. Indones.*, vol. 1, no. 3, pp. 24–28, 2022, doi: 10.55542/jppmi.v1i3.229.
- [19] N. N. A. Said and H. Purnama, "Pembuatan Bioetanol dari Limbah Kulit Kopi Arabika dan Robusta dengan Variasi Waktu Fermentasi," *Proceeding of The URECOL*, vol. 11, pp. 220–228, 2020, [Online]. Available: http://repository.urecol.org/index.php/proceeding/article/view/921
- [20] S. Aprilyanti, F. Suryani, and I. Pratiwi, "Optimasi Waktu Hidrolisis dan Volume Enzim Pada Proses Hidrolisis Enzimatis Selulosa Jerami Padi," *Pros. Semin. Nas. Has. …*, pp. 78–86, 2019, [Online]. Available: https://core.ac.uk/download/pdf/270229381.pdf
- [21] Zulnazri, A. P. Putri, R. Dewi, S. Bahri, and Sulhatun, "Karakterisasi Glukosa sebagai Bahan Baku Bioetanol yang Diproduksi dari α-Selulosa Berbasis Limbah Kulit Kopi Arabika," *Teknol. Kim. Unimal*, vol. 11, no. 1, pp. 102–111, 2022.
- [22] Jasman and R. M. Ahmad, "Pengaruh Jenis Perlakuan Awal terhadap Konsentrasi Bioetanol Hasil Hidrolisis dan Fermentasi Tongkol Jagung menggunakan Trichoderma reesei dan Saccharomyces cerevisiae," *J. Beta Kim.*, vol. 1, no. 2, pp. 25–34, 2021.
- [23] S. Y. M. Nggai, S. M. D. Kolo, and Y. Sine, "Pengaruh Perlakuan Awal Hidrolisis Ampas Sorgum (Sorghum Bicolor L.) Terhadap Fermentasi Untuk Produksi Bioetanol Sebagai Energi Terbarukan," *ALCHEMYJournal Chem.*, vol. 10, no. 2, pp. 33–40, 2022, doi: 10.18860/al.v10i2.13501.
- [24] P. Widyastuti, "Pengolahan Limbah Kulit Singkong Sebagai Bahan," *J. Kompetensi Tek.*, vol. 11, no. 1, pp. 41–46, 2019.
- [25] S. M. D. Kolo, J. Presson, and P. Amfotis, "Produksi Bioetanol sebagai Energi Terbarukan dari Rumput Laut Ulva reticulata Asal Pulau Timor," *ALCHEMY J. Penelit. Kim.*, vol. 17, no. 2, p. 159, 2021, doi: 10.20961/alchemy.17.2.45476.159-167.
- [26] L. Efiyanti, S. Sutanto, N. Hakimah, D. A. Indrawan, and G. Pari, "Karakterisasi Dan Potensi Katalis Karbon Aktif Tersulfonasi Limbah Kayu Pada Reaksi Hidrolisis Sekam Padi Menggunakan Microwave," *J. Penelit. Has. Hutan*, vol. 37, no. 2, pp. 67–80, 2019, doi: 10.20886/jphh.2019.37.2.67-80.
- [27] N. E. Wardani, W. A. Subaidah, and H. Muliasari, "Ekstraksi dan Penetapan Kadar Glukomanan dari Umbi Porang (Amorphophallus muelleri Blume) Menggunakan Metode DNS," *J. Sains dan Kesehat.*, vol. 3, no. 3, pp. 383–391, 2021, doi: 10.25026/jsk.v3i3.574.
- [28] S. N. Santi and T. Widyaningrum, "Produksi Bioetanol dari Limbah Batang Kelapa

Sawit (Elaeis guineensis) Menggunakan Zymomonas mobilis dengan Perlakuan Crude Enzim Trichoderma reesei dan Aspergillus niger," *J. Biolokus*, vol. 5, no. 1, pp. 18–23, 2022.

- [29] M. S. Fajri, M. A. Satrio, L. I. Utami, and K. N. Wahyusi, "Produksi Gula Cair dengan Proses Hidrolisis Asam dengan Bahan Pati Singkong," *ChemPro*, vol. 3, no. 1, pp. 58– 64, 2022, doi: 10.33005/chempro.v3i1.157.
- [30] H. P. Koapaha, "Issn 2303-1174," vol. 10, no. 1, pp. 1127–1146.
- [31] E. Sukara, H. Ambarsari, and A. Hartono, "PENGARUH KONSENTRASI NITRAT DAN KONSENTRASI ISOLAT SEDIMEN KOLAM IKAN LELE (Clarias SP.) PADA PROSES DENITRIFIKASI," *Biodidaktika, J. Biol. Dan Pembelajarannya*, vol. 14, no. 1, pp. 22–28, 2019, doi: 10.30870/biodidaktika.v14i1.4840.
- [32] A. S. P. PRASMONO and Atina Ahdika, "Analisis Regresi Berganda pada Faktor-Faktor yang Mempengaruhi Kinerja Fisik Preservasi Jalan dan Jembatan Di Provinsi Sumatera Selatan," *Emerg. Stat. Data Sci. J.*, vol. 1, no. 1, pp. 47–56, 2023, doi: 10.20885/esds.vol1.iss.1.art6.
- [33] K. Anwar, F. Istiqamah, and S. Hadi, "Optimasi Suhu dan Waktu Ekstraksi Akar Pasak Bumi (Eurycoma longifolia jack.) Menggunakan Metode RSM (response surface methodology) dengan Pelarut Etanol 70%," *J. Pharmascience*, vol. 8, no. 1, p. 53, 2021, doi: 10.20527/jps.v8i1.9085.
- [34] B. I. Widyananda and E. Komalasari, "Formulasi Minuman Fungsional Ekstrak Bawang Hitam dengan Penambahan Lemon dan Madu Menggunakan Response Surface Methodology," vol. 43, no. 1, pp. 94–104, 2023.
- [35] H. Kulit *et al.*, "Hidrolis Kulit Buah Kopi Oleh Kapang Pestalotiopsis sp . VM 9 Serta Pemanfaatan Hidrolisatnya Sebagai Medium Produksi Protein Sel Tunggal Saccharomyces cerevisiae ( Hydrolysis Coffea Pulp Using Extracelluler Enzymes of Pestalotiopsis sp. VM 9 and Utiliza," no. 2013, 2018.