



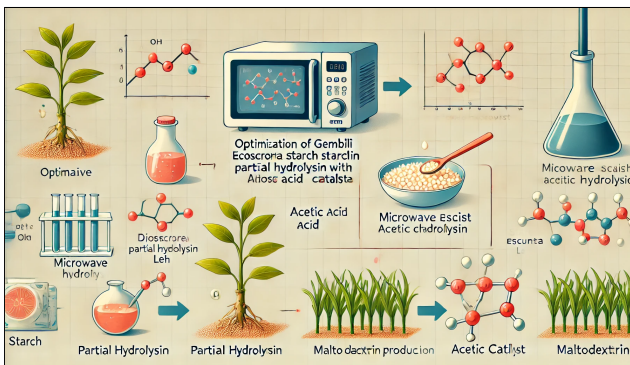
RESEARCH ARTICLE

Optimization of gembili (*Dioscorea esculenta* L.) starch partial hydrolysis in maltodextrin production with microwave assist using acetic acid catalyst

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OBJECTIVES The purpose of this study was to determine the optimal conditions for partial hydrolysis of gembili starch in the maltodextrin production. Novelty of this research is the use of acetic acid as a substitute for commonly used acids and microwaves for process efficiency. **METHODS** The process of maltodextrin production includes raw material pretreatment, gelatinization, liquefaction, drying and analysis. Variations in liquefaction time (30, 40, 50 min), microwave power (300, 400, 500 W) and acetic acid concentration (14, 15, 16 %) were used as independent variables. **RESULTS** The equivalent dextrose analysis results were 9.389 ± 0.042 to $18.980 \pm 0.201\%$, the density analysis results were 1.059416 to 1.107796 g/ml and viscosity analysis results were 0.430554 to 0.974663 cP. This study produces that 96.705% of the total variability in response can be explained in the regression equation. **CONCLUSIONS** Critical value of this study estimated dextrose equivalent value of maltodextrin produced of 16.636% and the validation of it is $16.254 \pm 0,074\%$.

KEYWORDS acetic acid; gembili starch; microwave; optimization; partial hydrolysis

1. INTRODUCTION

Maltodextrin is a white powder that is odorless and has a slightly sweet taste (AHOUEI et al. 2019; Pai et al. 2015). Maltodextrin is an intermediate product from starch hydrolysis using acid or enzyme catalyst (Paramita et al. 2012). Maltodex-

trin is widely used in industry, one of which is as a thickener or stabilizer in the food industry (Park and Walsh 2019; Triyono et al. 2017). Maltodextrin production uses a partial hydrolysis process because in the reaction of breaking the glycosidic bond of amylopectin compound, OH^- ions still remain. This happens when the α -1,4-glycoside bond that connects the main chain is broken so that two ions finish reacting. Meanwhile, the α -1,4-glycoside bond side chain is not interrupted, leaving 3 sides that will bind 2 H^+ ions and 1 OH^- ion, and causing the OH^- ion to not be completely reacted (Lupo et al. 2020; Yáñez-Alarid et al. 2020; Roat-Malone 2007). The process of hydrolysis requires catalyst so that the process can run quickly. Acid catalyst are more widely used in hydrolysis processes because the process is easy and simple (Anggoro et al. 2021; Muhaimin and Sudiono 2017; M et al. 2016). The acid that is often used in the hydrolysis process is a strong acid. However, when these acids are used in maltodextrin production as a food ingredient, it is very risky and dangerous. Moreover, if the maltodextrin product is commercialized, it will be exposed to food safety issues. Therefore, acetic acid is used where this acid has been widely consumed (Harianja et al. 2015; Lourenço et al. 2019; Perdana 2018).

Besides acid catalyst, the hydrolysis process is also affected by heating (M.A. et al. 2019; Priatna et al. 2021; Sun et al. 2015). Conventional heating in the hydrolysis process produces low yield, a long process and expensive costs. Besides conventional heating, microwave irradiation can also be used as a heater. Microwave irradiation will cause the reaction rate to increase by 5-100 times. Therefore, the hydrolysis with microwave assistance is a new approach and can address previous problems (Li and Xu 2017; Okada and Maeda 2021; Rokhati et al. 2020).

The raw material in this research uses gembili (*Dioscorea esculenta* L.) starch. Gembili starch is used because of its great potency and high starch content, with an amylopectin content of 85,8%. One of the regions with great gembili potential is Papua, Indonesia with its production reaching 70 tons/ha/year (Fera and Masrikhiyah 2019; Latifah and Prahardini 2020; Sabda et al. 2019).

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2. MATERIALS AND METHOD

2.1 Materials

The details of the materials in the research series can be seen as follows: Gembili (*Diocorea esculenta* L.) from Indonesian local farmers, CH₃COOH 100% (Merck, Germany), Fehling's solution A (Merck, Germany), D-Glucose Anhydrous (Merck, Germany), and Methylene Blue (Merck, Germany).

The sample preparation process begins with stripping the skin of gembili. After that, gembili are washed until all dirt disappears. Next, soaking is carried out using 0,3% CaO to remove the sap. Then, gembili are cut into pieces and blended. The slurry of gembili is taken as much as 250 g and dissolved in 2 L water. Then it is precipitated and resulting precipitated is dried in the sun to dry. After drying, the starch of gembili is sifted using a 50 mesh sieve (Rukmini and Santosa 2019).

2.2 Procedures

2.2.1 Gelatinization process

The gelatinization process in this study was carried out by adding acetic acid solution. As much as 30 g of dried gembili (*Dioscorea esculenta* L.) starch was put into a beaker glass and added 300 ml of acetic acid solution, then stirred until the starch dissolved. Next, gelatinization of the mixture was carried out using microwave assistance for 7,5 min.

2.2.2 Liquefaction process

The liquefaction process in this study was carried out by heating using mantle heaters accompanied by stirring at a temperature of 95°C. This liquefaction process where the acid will enter the the pores of the material, combine with the water contained in the material, will breaks down starch molecules (Rahmawati et al. 2020).

2.2.3 Determine of physical propertise (density and viscosity)

Density determination was determined by the relationship between the mass of the pycnometer and the volume of the pycnometer (Pentury et al. 2013), namely Equation 1.

$$\rho_{\text{maltodextrin}} = \frac{m_b - m_a}{v_c} \quad (1)$$

Where, $\rho_{\text{maltodextrin}}$ = density of maltodextrin solution (g/mL), m_a = mass of empty pycnometer (g), m_b = mass of filled pycnometer (g), v_c = volume of pycnometer (mL).

The viscosity value, was determined by the relationship between the flowing time and density (Marta et al. 2017), namely Equation 2.

$$\frac{\eta_1}{\eta_2} = \frac{\rho_1 \cdot t_1}{\rho_2 \cdot t_2} \quad (2)$$

where,

1. η_1 = the viscosity value of the sample (poise),
2. η_2 = the viscosity value of distilled water (poise),
3. ρ_1 = the density of the sample (g/ml),
4. ρ_2 = the density of the distilled water sample (g/ml),

5. t_1 = the sample flowing time (s), t_2 = distilled water sample flowing time (s).

2.2.4 Drying process

The resulting liquid maltodextrin was then dried in an oven at 130°C for 5 hours. After drying, the maltodextrin was crushed and then sieved to form maltodextrin powder.

2.2.5 Determine of dextrose equivalent

Dextrose equivalent (DE) is a quantity that expresses the total value of starch reducer or starch modified products. Fehling volumetric method was used for dextrose equivalent analysis (Shi and Jeffcoat 2000). Find the fehling factor value by dissolving 2.5 g of anhydrouse glucose with 1000 ml of distilled water. Then, 50 ml of distilled water was put into the Erlenmeyer and added 5 ml of fehling A and fehling B. Then, boil the mixture, and add 3 drops of methylene blue. The solution is titrated with the previous glucose solution until it turns reddish brown. Record the titrant requirement and the fehling factor (FF) by Equation 3.

$$FF = \frac{\text{Titrant needs (ml)} \times \text{mass of glucose (gr)}}{1000\text{ml}} \quad (3)$$

After determining the FF value, 5 g of maltodextrin powder is taken and dissolved in 100 ml of aquadest. The solution is put into the burette. Then, 50 ml of distilled water is put into the Erlenmeyer and 5 ml of fehling A and B are added. After that, the mixture is boiled and 3 drops of methylene blue are added. The solution is titrated with the previous maltodextrin titrate solution until it turns reddish brown. Record the titrant requirement and then calculate the dextrose equivalent value by Equation 4.

$$DE = FF \times \frac{100}{\text{Concentration of maltodextrin solution } \left(\frac{\text{gr}}{\text{ml}}\right) \times \text{titrant needs (ml)}} \quad (4)$$

2.2.6 Response surface methodology statistical analysis

The response surface experiment is designed by applying the Central Composite Design of the alpha for orthogonality (Stattistica 10 by Statsoft, Europe). The independent variables of the process are acetic acid concentration (X_1), microwave power (X_2), and liquefaction time (X_3). Each optimized variable was coded at five levels, namely $-\alpha$, -1 , 0 , $+1$, $+\alpha$. The response obtained was the value of dextrose equivalent.

3. RESULTS AND DISCUSSION

The results of density, viscosity and dextrose equivalent analysis are shown in table 2. After that, the analysis was carried out using response surface experiment (Stattistica 10 by Statsoft, Europe).

3.1 Results of density analysis

The results of density analysis are shown in table 2, where the lowest value of 1.059 g/ml occurs at 15% acetic acid concentration, 400 W of microwave power, and 23.18 minute of liquefaction time. Meanwhile, the highest value of 1.108 g/ml occurs at 14% of acetic acid concentration, 400 W of microwave power and 50 minutes of liquefaction time.

TABLE 1. Central composite design.

Independent Variables	Coded variables levels				
	- α	-1	0	+1	+ α
Acetic acid concentration (%)	13.31	14	15	16	16.68
Microwave power (W)	231.82	300	400	500	568.18
Liquefaction Time (min)	23.18	30	40	50	56.81

The most significant factors affecting the density are the duration of stirring. Stirring is an attempt to create movement of the stirred material (dispersed solid particles) in a solvent so that it can dissolve completely. The more homogeneous a solution, the more solute mass is dissolved in each volume of solvent, which will increase the density value (Komal Patel and Ingle 2019; Seager et al. 2018).

3.2 Results of viscosity analysis

The results of viscosity analysis are shown in table 2, where the lowest value of 0.431 cP occurs at 15% of acetic acid concentration, 568.18 W of microwave power and 40 minute of liquefaction time. Meanwhile, the highest value of 0.975 cP occurs at 15% of acetic acid concentration, 400 W of microwave power and 56.82 minute of liquefaction time.

The viscosity value is related to hydrolysis time, which is also related to the results of dextrose equivalent value. This is because the higher the dextrose equivalent value, the shorter the chain of the starch compounds produced from the hydrolysis process. This effect automatically occurs because the cutting of the amylose and amylopectin chains becomes shorter, resulting in a decrease in viscosity (Ikeda et al. 2022; Laga et al. 2018)

3.3 Results of dextrose equivalent analysis

The results of dextrose equivalent analysis are shown in table 2, where the lowest value of 9.389 ± 0.042 occurs at 15% of acetic acid concentration, 400 W of microwave power and 23.18 minute of liquefaction time. Meanwhile, the highest value of 18.980 ± 0.201 occurs at 15% of acetic acid concentration, 568.18 W of microwave power and 40 minute of liquefaction time.

Based on the dextrose equivalent results in Table 2, it is shown that the dextrose equivalent (DE) of maltodextrin produced ranges from 9-18. This indicates that the resulting product is included in the maltodextrin group because the DE value is still in the range of DE maltodextrin values of 3-20 (Saavedra-Leos et al. 2015; Xiao et al. 2022).

3.4 Results of response surface methodology statistical analysis

The dextrose equivalent data from the experiment were processed using the RSM method. RSM is one of the empirical statistical methods used to analyze multiple regression and can be used in solving multivariable equations simultaneously using multivariable-quantitative data (Damayanti et al. 2021; Korde et al. 2021; Yulianto et al. 2018).

TABLE 2. Central composite design experiment and experimental results.

Run	Experimental Design			Experimental Results		
	Acetic Acid Concentration (%) (X1)	Microwave Power (W) (X2)	Liquefaction Time (min) (X3)	Density (g/ml)	Viscosity (cP)	Dextrose Equivalent (%) (Y)
1	14	300	30	1.107	0.819	10.586 ± 0.054
2	14	300	50	1.108	0.687	12.018 ± 0.106
3	14	500	30	1.102	0.727	11.613 ± 0.037
4	14	500	50	1.099	0.626	15.333 ± 0.131
5	16	300	30	1.098	0.593	15.408 ± 0.066
6	16	300	50	1.087	0.467	16.690 ± 0.204
7	16	500	30	1.090	0.556	16.044 ± 0.071
8	16	500	50	1.093	0.535	16.425 ± 0.151
9	13.32	400	40	1.095	0.734	12.040 ± 0.040
10	16.68	400	40	1.102	0.672	16.127 ± 0.072
11	15	231.82	40	1.102	0.452	17.939 ± 0.089
12	15	568.18	40	1.104	0.431	18.980 ± 0.201
13	15	400	23.18	1.059	0.975	9.389 ± 0.042
14	15	400	56.82	1.069	0.652	13.161 ± 0.095
15	15	400	40	1.094	0.591	15.073 ± 0.125
16	15	400	40	1.085	0.586	15.720 ± 0.119

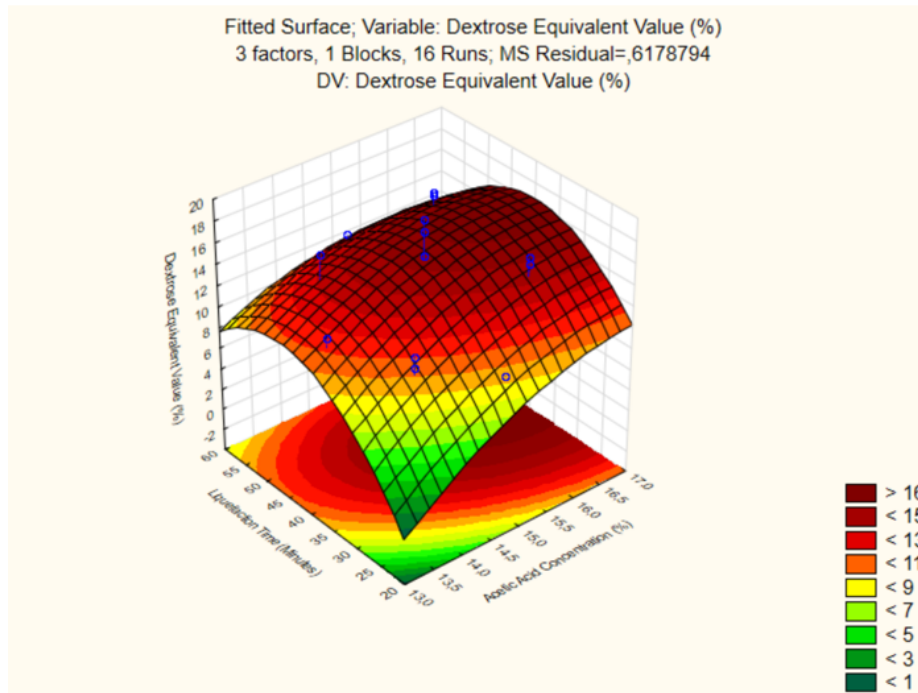


FIGURE 1. Contour plot of RSM response surface on the effect of acetic acid concentration and liquefaction time on DE value.

3.4.1 Effect estimate

The accuracy of a model used in research can be evaluated based on the correlation coefficient R^2 value. The R^2 value provides a measurement of how the experimental variables and their interactions can explain differences in variability in the observed response values. The R^2 value is in the range of 0-1, where if the R^2 value is closer to 1, it indicates that the model used is good at predicting the response (Lu et al. 2023; Paramita et al. 2016; Yulianto et al. 2018) This study produces a coefficient of determination is 0.96705. It can be interpreted that 96.705% of the total variability in response can

be explained by the regression equation (Equation 5).

$$15.2512 + 3.20565X_1 - 1.06498X_1^2 + 0.94638X_2 + 2.02931X_2^2 + 1,92691X_3 - 3.05105X_3^2 - 0.99294X_1X_2 - 0,87167X_1X_3 + 0.34698X_2X_3 \quad (5)$$

3.4.2 Response surface contour plot

In figure 1, the surface contour plot of the response to the effect of acetic acid concentration and liquefaction time is presented. Higher acid concentration and longer liquefaction time result in a higher dextrose equivalent value (Kong

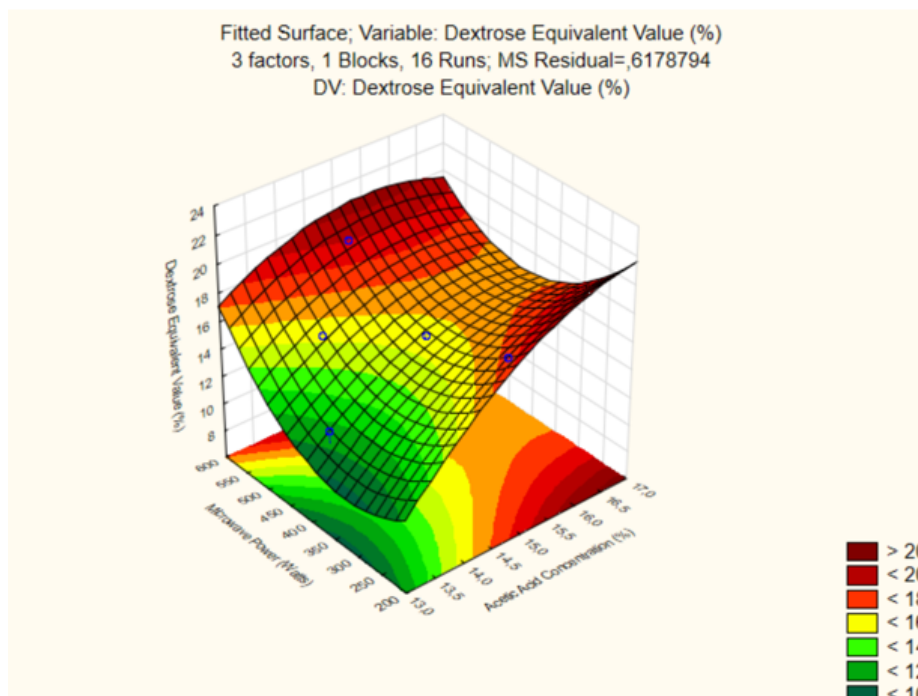


FIGURE 2. Contour plot of RSM response on the effect of acetic acid concentration and microwave power on DE value.

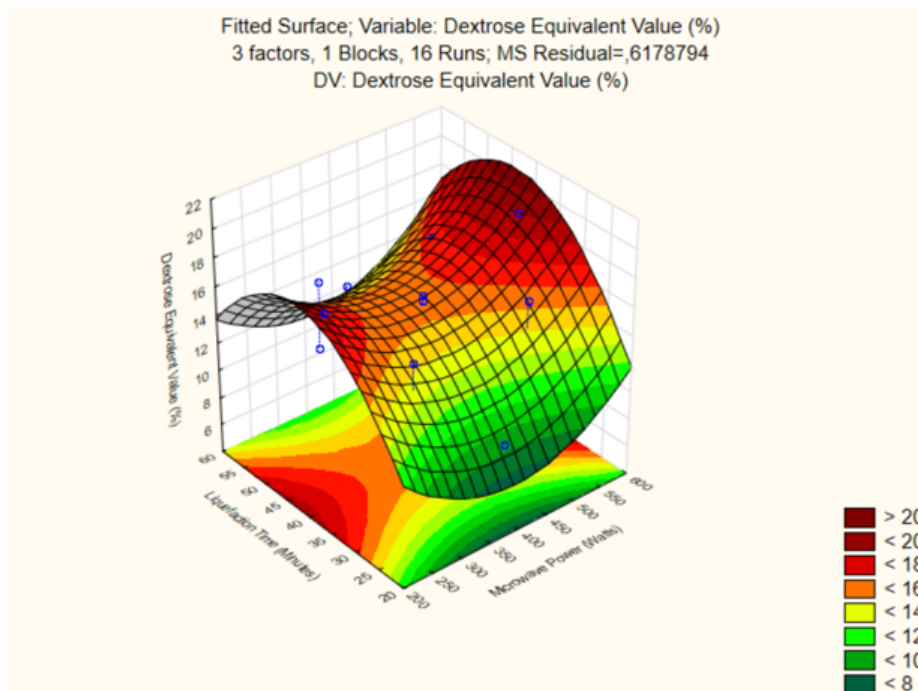


FIGURE 3. Contour plot of RSM response surface on the effect of microwave power and liquefaction time on DE value.

et al. 2018; Laga et al. 2020; Santosa and Handayani 2014). The higher concentration of the catalyst will decrease the activation energy so that the hydrolysis process will be faster (Muhaimin and Sudiono 2017; Rahmawati et al. 2020). The faster of hydrolysis process will break the chain of amylose and amylopectin compounds and it will affect the dextrose equivalent value. The shorter the chains of modified amylose and amylopectin compounds are related to the degree of polymerization of the final product (Subroto 2020; Vargas-Campos et al. 2023).

In figure 2, the surface contour plot of the response to the effect of acetic acid concentration and microwave power is

presented. The higher of microwave power will increase the heating temperature, so it will increase the effectiveness of hydrolysis (Fu et al. 2016; Jiang et al. 2023). This is because the greater of the power used to generate microwaves, it will make the greater of electric field strength. If the electric field strength is greater, will generate microwave amplitude is also greater. The rotational speed of the polar molecules has a linear relationship to the microwave amplitude. The amplitude greater, the polar molecules rotate will faster, so heat forming is faster (Cheng et al. 2022; Rosyida Permatasari, M. Sjahrul Annas 2015; Sujana et al. 2020).

In figure 3, the surface contour plot of the response to

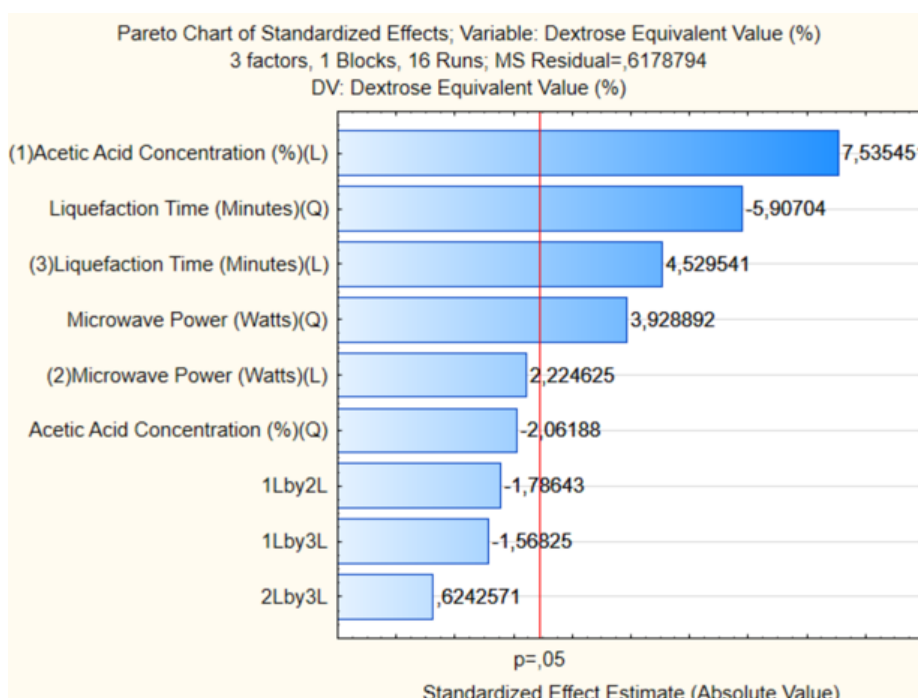


FIGURE 4. Pareto diagram of variable influence on dextrose equivalent value of maltodextrin.

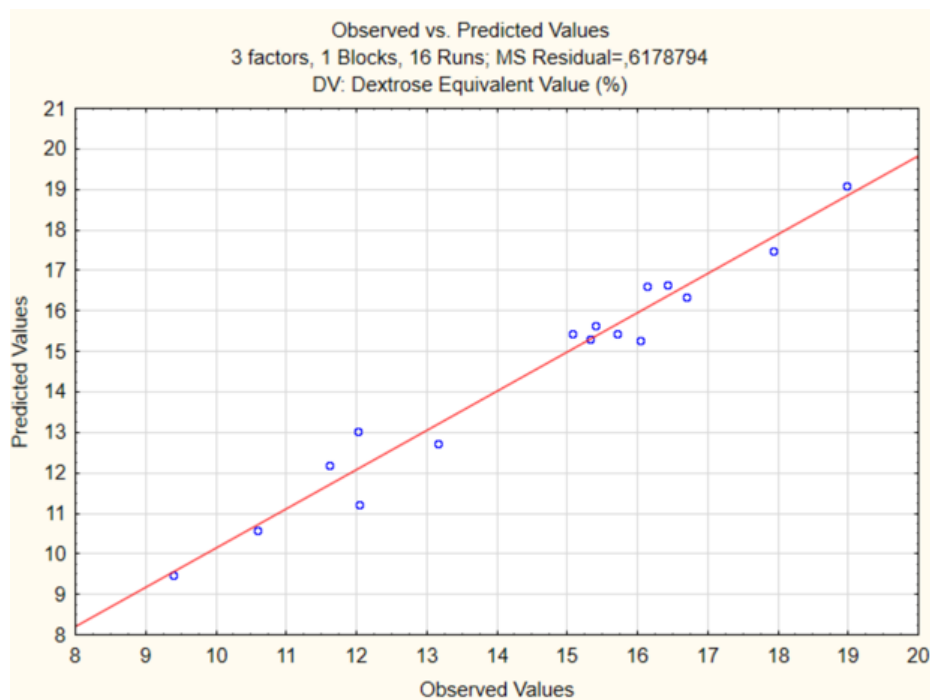


FIGURE 5. Comparison of experimental data and estimated dextrose equivalent values of maltodextrin.

the effect of microwave power and liquefaction is presented. The higher of gelatinization temperature makes the starch bubble faster and break easily so that the bonds between glucose units from amylose and amylopectin stretch more and are easy to break (Feng et al. 2020; Sobini et al. 2022). It will produce the chains of amylose and amylopectin compounds that are shorter. This affects the liquefaction time because it will increase the affectiveness during the liquefaction process (Arif et al. 2019; Gui et al. 2021).

3.4.3 Pareto diagram

In figure 4, a pareto diagram showing which variables were most influential in the experiment is presented. The most influential independent variable in the partial hydrolysis of gembili starch to produce maltodextrin is the acetic acid concentration (L), liquefaction time (Q), liquefaction time (L) and microwave power (Q), seen from the independent parameter value which is more than 0.5 as the p value (Endy Yulianto et al. 2022, 2020).

3.4.4 Comparison data runs vs dextrose equivalent analysis

The relation between the predicted values and the model results obtained from the experiment is presented in Figure 5. The plot formed in the figure shows the experimental

data where it can be seen that there are deviations at several points from the estimated value. However, the deviation between these values shows a relatively good correlation because the resulting research data is close to the linear line of the estimated value. Regression coefficients are clarified using pareto diagrams and ANOVA for each influential variable (Lu et al. 2023; Nisa and Paramita 2021).

3.4.5 Predicted and validation value of dextrose equivalent analysis

Parameter optimization for partial hydrolysis of gembili (*Dioscorea esculenta* L.) starch using acetic acid and microwave assistance on acetic acid concentration, microwave power, and liquefaction time was carried out by determining the critical values shown in Table 3. The critical value for dextrose equivalent optimization obtained through RSM analysis is saddlepoint-shaped, saddlepoint-shaped occur which are characterized by a contour plot that forms a horseshoe (Lu et al. 2023; Sofyan et al. 2018), with an estimated dextrose equivalent value of maltodextrin produced of 16.636 which will be achieved at an acetic acid concentration of 16.41%, microwave power of 410.12 W, and liquefaction time of 41.20 min. Based on the critical value experiment that has been carried out, the dextrose equivalent obtained is 16.254 ± 0,074. The results obtained from the critical value experiment are close to the predicted value of RSM.

TABLE 3. Predicted and validation value of dextrose equivalent analysis.

Factor	Minimum Value	Critical Value	Maximum Value
Acetic acid concentration (%)	13.32	16.41	16.68
Microwave power (W)	231.82	410.12	568,18
Liquefaction time (min)	23.18	41.20	56.82
Predicted value of dextrose equivalent	16.636		
Validation value of dextrose equivalent	16.254 ± 0.074		

TABLE 4. Analysis variance of gembili (*Dioscorea esculenta* L.) starch partial hydrolysis.

Factor	SS	Df	MS	F
Acetic acid concentration (%) (L)	35,0851	1	35,08506	56,78302
Acetic acid concentration (%) (Q)	2,6268	1	2,62681	4,25133
Microwave Power (W) (L)	3,0579	1	3,05786	4,94896
Microwave Power (W) (Q)	9,5377	1	9,53770	15,43619
Liquefaction Time (min) (L)	12,6769	1	12,67687	20,51674
Liquefaction time (min) (Q)	21,5598	1	21,55976	34,89315
1L by 2L	1,9719	1	1,97186	3,19134
1L by 3L	1,5196	1	1,51962	2,45942
2L by 3L	0,2408	1	0,24079	0,38970
Error	3,7073	6	0,61788	
Total SS	112,5234	15		142.86985

3.4.6 Analysis of variance

The response surface model in the analysis of variance (ANOVA) form is shown in Table 4. ANOVA is required to test the significance and adequacy of the model. The Fisher variance ratio or F value is a valid statistical measure of how well a factor explains the variation in the mean data, and the estimated effect of the factor is real. The greater the F value, the more it indicates uniformity (Malla et al. 2023; Paramita et al. 2016; Xie et al. 2023) The ANOVA of the regression model shows that it exhibits a significant correlation, as evident from the F value of the Fisher test ($F_{\text{model}} = 142.86985$).

4. CONCLUSIONS

The response surface methodology is used to optimize the dextrose equivalent value of maltodextrin production from gembili starch. Acetic acid concentration (%) (X_1), microwave power (W) (X_2) and liquefaction time (min) (X_3) are used as independent variables. The results indicate that the acetic acid concentration (L), liquefaction time (Q), liquefaction time (L) and microwave power (Q) are the most significant factors in this process. This study shows that 96.705% of the total variability in response can be explained in the regression equation. The Critical value of this study estimates the dextrose equivalent value to be maltodextrin produced of 16.636 which will be achieved at an acetic acid concentration of 16.41%, microwave power of 410.12 W, and liquefaction time of 41.20 min. The validation of it is $16.254 \pm 0,074$.

5. ACKNOWLEDGEMENTS

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