Optimization of Sorghum Starch (*Sorghum bicolor* L.) Partial Hydrolysis using Microwave-Assisted Acetic Acid Catalyst

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ABSTRACT

Hydrolysis process is relatively slow, requiring the use of catalyst and microwave assistance to accelerate the reaction. There fore, this study aimed to determine the optimum conditions for partial hydrolysis of sorghum starch (*Sorghum bicolor* L.) using acetic acid as a microwave-assisted catalyst to produce maltodextrin. The experiment was carried out in several stages, namely gelatinization, liquefaction, and drying, while Response Surface Method (RSM) was used for variable design and data analysis. Hydrolysis process was carried out with three independent variables, including acetic acid concentration (9%, 12%, and 15%), microwave power (300 watts, 400 watts, and 500 watts), and liquefaction time (35 minutes, 45 minutes, and 55 minutes). The results showed that the highest DE (Dextrose Equivalent) value of maltodextrin was found at 17.04% acid concentration, 400 watts microwave power, and 45 minutes liquefaction time, valued at 18.921 ± 0.099. The optimum dextrose equivalent of 17.763 would be achieved at 16.879% acetic acid concentration, 390.233 watts of microwave power, and 47.055 minutes of liquefaction time. This study introduces the innovation of using acetic acid as a catalyst and microwave assistance during the gelatinization stage.

Keywords: Acetic acid; dextrose equivalent; microwaves; partial hydrolysis; sorghum starch

INTRODUCTION

Maltodextrin is a saccharide polymer and nutritious substance in the form of white odorless powder with a sweet taste. Furthermore, it is widely used as stabilizer in the food industry (Herlina et al., 2017) and thickener in the nanoemulsion process (Nawangsasi et al., 2018). To break down polysaccharides (complex sugars) including maltodextrin, hydrolysis process is generally used by binding with water to produce a new simpler substance (Aniriani et al., 2018). This process occurs in the form of partial and total hydrolysis, by splitting water (H_2O) into H^+ and OH^- ions. During the reaction, partial hydrolysis is indicated by the presence of unreacted ions, while total hydrolysis occurs when both ions have reacted (Safitri et al., 2018).

The formation of maltodextrin requires partial hydrolysis, as the glycoside bond in the amylopectin compound passes through incomplete breakdown due to the presence of OH- ions. This phenomenon occurs because the bond connecting the α-1,4-glycoside main chain to the α-1,6-glycoside side chain is released, with H^+ ions binding to CH_2 compound to inhibit the complete reaction of OH- ions. Although water can facilitate hydrolysis process, the effectiveness is limited due to the slow reaction rate, indicating the need for

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catalyst. In this study, acetic acid was used as catalyst to accelerate hydrolysis process, ensuring safety for consumption when left in the resulting maltodextrin product. Additionally, this inorganic acid was used due to cost-effectiveness and availability compared to others (Listiani et al., 2016).

Foodstuffs that contain high carbohydrates have the potential to produce starch. Among these foodstuffs, sorghum (*Sorghum bicolor* L.) with high starch content of approximately 80.42% can be used as a base material for hydrolysis to be converted into maltodextrin. In addition catalysts, heating also plays an important role in accelerating the reaction process in hydrolysis (Priatna et al., 2021). However, conventional heating produces low yields and requires a prolonged processing time, leading to high production costs. To address these challenges, microwave irradiation can be used as an alternative solution, accelerating the reaction rate by 50-100 times (Kolo & Sine, 2019). Consequently, hydrolysis process with microwave heating represents a new approach that offers a potential solution to previous problems.

METHODS

Materials

The materials used in this study included sorghum flour obtained from Semarang, Indonesia. Additionally, acetic acid 99%, anhydrous D-glucose, Fehling A, Fehling B, and methylene blue indicator were purchased from Merck (Darmstadt, Germany), while distilled water was obtained from CV Indrasari (Semarang, Indonesia). The equipments used included Microwave Oven with Rated output power of 800 watt (AB Electolux publ, Stockholm, Sweden), Heating mantle, magnetic stirrer, oven, and a series of titration tools such as burettes, stative clamps, and Erlenmeyer.

Methods

Experimental design

The experiment was designed by using Response Surface Method (RSM), offering improved result reproducibility and process optimization, along with a detailed perspective for the development of predictive models. Table 1 shows the experiment design with 16 runs and independent variables of acetic acid concentration, microwave power, and liquefaction time.

Gelatinization of sorghum starch

The gelatinization process in this study was carried out by adding acetic acid with a concentration according to the specified variables. A total of 30 g dried sorghum starch was put into a glass container, followed by the addition of 300 ml acetic acid solution into the glass container, and stirred until the starch dissolved. Subsequently, the mixture was gelatinized using a microwave for 7 minutes of duration followed by liquefaction process.

Table 1. The experiment design of optimization of sorghum starch partial hydrolysis

Experiment	Acetic acid concentration (%)	Microwave power (watt)	Liquefaction time (min)
1	9.00	300.00	35.00
2	9.00	300.00	55.00
3	9.00	500.00	35.00
4	9.00	500.00	55.00
5	15.00	300.00	35.00
6	15.00	300.00	55.00
7	15.00	500.00	35.00
8	15.00	500.00	55.00
9	6.95	400.00	45.00
10	17.05	400.00	45.00
11	12.00	231.82	45.00
12	12.00	568.18	45.00
13	12.00	400.00	28.18
14	12.00	400.00	61.82
15	12.00	400.00	45.00
16	12.00	400.00	45.00

Liquefaction of sorghum starch

Liquefaction process is carried out by heating according to the predetermined temperature variable of 95 °C. This process was accompanied by stirring using a magnetic stirrer according to the specified time variable and liquid maltodextrin was analyzed for density and viscosity.

Analysis of density of maltodextrin solution

Analysis of liquid maltodextrin density was carried out by measuring the weight of an empty and a filled pycnometer. After obtaining the weight, density calculation was carried out using Equation 1 (Yuliawaty & Susanto, 2015).

$$\rho = \frac{Weight\ of\ Filled\ Pycnometer - Weight\ of\ Empty\ Pycnometer}{Volume\ of\ Pycnometer} \quad (1)$$

Viscosity analysis of maltodextrin solution

Viscosity analysis of maltodextrin solution was carried out using an Ostwald Viscosimeter by measuring the time required for the tested sample liquid to pass the predetermined limit on the tool. After obtaining the time required, viscosity of maltodextrin solution was determined through calculation using Equation 2 (Yuliawaty & Susanto, 2015).

$$\mu = \mu_0 \frac{t.\rho}{t_0.\rho_0} \quad (2)$$

where μ is viscosity of the sample liquid (cp), μ0 is viscosity of the comparison liquid (cp), t is the flow time of the sample liquid (s), t0 is the flow time of the comparison liquid (s), ρ is the density of the sample liquid (g/mL), and ρ0 is the density of the comparison liquid (g/mL).

Drying maltodextrin solution

Maltodextrin solution produced was dried using an oven with a temperature of 130°C for 180 minutes. Subsequently, the dried sample was crushed with a blender and sieved to form maltodextrin powder. The yield produced was calculated and Dextrose Equivalent (DE) test was carried out to determine maltodextrin quality.

Analysis of Dextrose Equivalent (DE)

Analysis of DE value was carried out by titration method according to the procedure in previous study (Meriatna, 2013). Determination of fehling factor value was performed by dissolving 2.5 g anhydrous glucose with aquadest to a volume of 1000 mL. Subsequently, 50 ml aquadest was put into an erlenmeyer, followed by the addition of 5 ml each of Fehling A and B. The mixture was brought to a boil, added with 3 drops of methyl blue indicator, and titrated with the previous glucose solution until reddish brown. The volume of titrant required was recorded, allowing for the calculation of Fehling Factor (FF) value using Equation 3.

$$FF = \frac{Titrant\ required\ (mL) x\ weight\ of\ glucose\ (g)}{1000\ mL} \quad (3)$$

After obtaining FF value, 5 g of the resulting maltodextrin powder was taken and dissolved in 100 mL aquadest. The solution was put into a burette and 50 mL aquadest was placed into an erlenmeyer, added with 5 mL each of Fehling A and B solution. Subsequently, the mixture was brought to a boil, added with 3 drops of methylene blue indicator, and titrated with maltodextrin solution until reddish brown and precipitate appeared. The volume of titrant required was recorded, allowing for the calculation of DE value by Equation 4.

$$DE = FF\ x\ \frac{100}{Concentration\ of\ maltodextrin\ solution\left(\frac{g}{mL}\right) x\ Titrant\ required\ (mL)} \quad (4)$$

RESULTS AND DISCUSSION

Density Analysis of Maltodextrin Solution

In this study, density analysis was carried out on the sample solution that had been liquefied. According to (Meriatna, 2013), the factor that affects density includes the length of time for stirring which occurs in the liquefaction section. Based on the results, the highest density value of 1,1092 g/mL was obtained at 12% acid concentration, 400 watts microwave power and 61,81 minutes liquefaction time. Meanwhile, the lowest value of 1,0672 g/mL was obtained at 12% acetic acid concentration, 400 watts microwave power, and 28 minutes liquefaction time. As described by Syamani et al. (2020), stirring facilitates the dispersion and dissolution of solid particles in a solvent, leading to increased homogeneous solution and density. Consequently, the highest density value obtained in this study was observed at 61.81 minutes of liquefaction time.

Analysis of Viscosity of Maltodextrin Solution

Viscosity analysis was conducted in the laboratory using an Ostwald Viscometer, expressing the size of friction in the fluid (Lumbantoruan & Yulianti, 2016). However, viscosity is influenced by the length of time liquefaction on the sample. In this study, the lowest viscosity of 0.339 cp was obtained at 12% concentration, 400 watts microwave power with a liquefaction time of 28.18 minutes. Meanwhile, the highest value of 0.498 cp was obtained from a concentration of 12%, 400 watts of microwave power with a liquefaction time of 61.81 minutes. During the hydrolysis process at the liquefaction stage, bond breaking (degradation) of complex carbohydrate compounds with long chains and high molecular weight occurred into simple compounds or monosaccharides with short chains and low molecular weight. Although liquefaction was carried out at 95°C, the degradation or polymerization resulted in a decrease in viscosity (Santosa & Handayani, 2012). This showed that the lowest viscosity value obtained at the longest liquefaction time of 61.81 minutes, was in accordance with the theory of viscosity reduction.

Analysis of Dextrose Equivalent (DE)

The measurement of DE value is important in the preparation of maltodextrin, serving as an indicator of

Table 2. The experiments of sorghum starch partial hydrolysis

Experiment	Yield (%)	Density (g/cm^3)	Viscosity (mPa.s)	Dextrose equivalent
1	96.50	1.070	0.4904	10.55±0.08
2	97.86	1.088	0.4206	11.18±0.07
3	96.55	1.071	0.4332	10.85±0.03
4	98.93	1.089	0.3525	10.88±0.03
5	95.20	1.0700	0.4136	15.41±0.07
6	97.86	1.0896	0.3428	16.92±0.16
7	96.77	1.0708	0.4428	16.13±0.07
8	98.92	1.0896	0.4016	16.38±0.20
9	96.63	1.0828	0.4088	7.52±0.03
10	98.55	1.0836	0.4189	18.92±0.10
11	96.33	1.0820	0.4085	16.39±0.33
12	98.85	1.0816	0.3889	16.78±0.16
13	95.25	1.0672	0.4989	13.90±0.11
14	99.59	1.1092	0.3390	13.16±0.05
15	95.96	1.0884	0.3914	15.11±0.13
16	98.26	1.0832	0.3993	15.41±0.07

hydrolyzed starch. Table 2 shows the experiments of sorghum starch partial hydrolysis with DE value.

Table 2 shows that DE results of maltodextrin produced ranged from 7-18. This shows that the resulting product is included in the maltodextrin group, as DE value varies between 3-20 (Meriatna, 2013).

Statistic Analysis of Response Surface Method (RSM)

DE data from the experiment was processed using RSM, an empirical statistical method capable of analyzing multiple regression and solving multivariable equations simultaneously with multivariable-quantitative data (Yohana et al., 2022). A response fit surface can be created from the experimental results in Figure 1, showing the relationship between acetic acid concentration, microwave power, and liquefaction time to maltodextrin DE. By applying multiple regression analysis to the experimental data, a second-order polynomial equation was obtained to represent the effect of the independent variables on maltodextrin DE as expressed in Equation 5.

$$Y = 15{,}34204 + 5{,}93958X_1 - 1{,}84178X_1^2 + 0{,}12420X_2 + 0{,}53635X_2^2 + 0{,}17444X_3 - 1{,}62366X_3^2 + 0{,}04613X_1X_2 + 0{,}27532X_1X_3 - 0{,}45979X_2X_3 \quad (5)$$

where X_1 is acetic acid concentration, X_2 is microwave power, and X_3 is liquefaction time.

The optimization process of partial hydrolysis of sorghum starch was carried out through 16 experiments, as shown in Table 1. In this study, the accuracy of model used was evaluated based on the correlation coefficient R^2 value, which served as an indicator of how the experimental variables and interactions can explain differences in variability in the observed response values. The R^2 value ranges from 0 to 1, where a value closer to 1 shows a good ability of model in predicting the response (Yohana et al., 2022). A high correlation between the predicted value and the observed response is shown by an R^2 value of more than 0.96. This shows that 96.47% of the variability in the response can be explained by the model.

The results of RSM contour plot show that high acetic acid concentration, microwave power, and liquefaction time increased DE value of maltodextrin. In this study, the optimal DE value of 18.92 was obtained at the highest acid concentration of 17.045% and liquefaction time of 61.81 minutes. The results are supported by Meriatna (2013) on the hydrolysis of sago starch into maltodextrin using hydrochloric acid, where the highest DE value of 5.24 was obtained at HCl concentration of 9% and hydrolysis time of 130 minutes. Marta et al. (2017) also investigated maltodextrin production from corn starch,

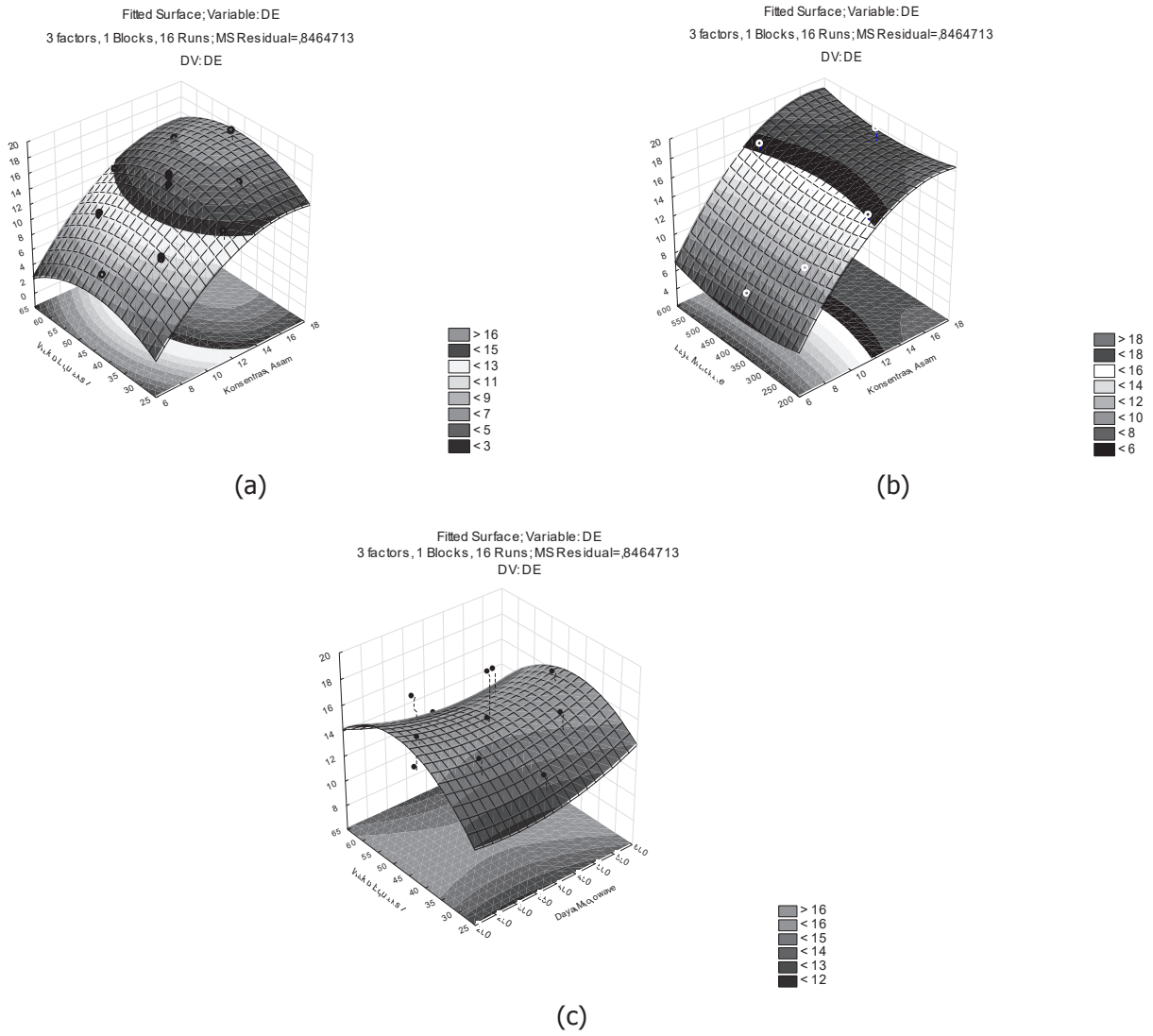

Figure 1. (a) Contour plot of RSM response surface on the effect of acetic acid concentration and liquefaction time on DE value; (b) Contour plot of RSM response on the effect of acetic acid concentration and microwave power on DE value; (c) Contour plot of RSM response surface on the effect of microwave power and liquefaction time on DE value

obtaining the highest DE value of 16.12 at a hydrolysis time of 35 minutes, and HCl concentration of 1.35%.

The relation between the predicted value and model results obtained from the experiment is shown in Figure 2(a). The plot formed in the figure showed the experimental data, indicating deviations at several points from the estimated value. However, these deviations had a relatively good correlation as the resulting study data were close to the linear line of the estimated value. Regression coefficients were clarified by using pareto diagrams and ANOVA for each influential variable (Paramita et al., 2016).

Pareto diagram showing the most influential variables in the experiment is presented in Figure 2(b). Based on the results, the most influential independent variable in partial hydrolysis of sorghum starch to produce maltodextrin is the acetic acid concentration. This showed that higher concentration of acetic acid resulted in greater production of maltodextrin and DE value.

The response surface model in analysis of variance (ANOVA) form is shown in Table 3, to determine the significance and adequacy of the model. Fisher variance ratio or F value is a valid statistical measure of how well a factor explains the variation in the mean data, indicating the real estimated effect. A previous study by (Paramita et al., 2016) has established that greater F value shows higher uniformity. In this study, ANOVA of the regression model showed significant correlation, as evident from the F value of the Fisher test (Fmodel = 160.4449).

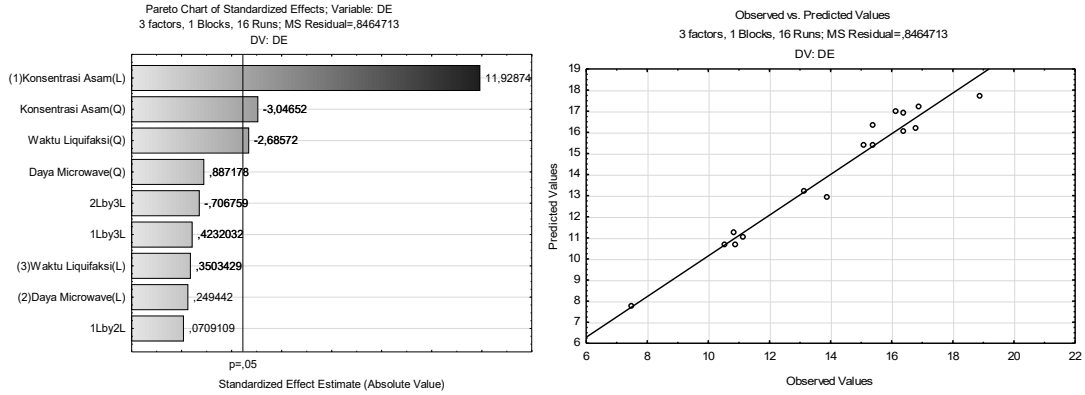

Figure 2. (a) Comparison of experimental data and estimated DE value of maltodextrin; (b) Pareto diagram of variable influence on DE value of maltodextrin

Table 3. Analysis of variance of sorghum starch hydrolysis polynomial equation model

Factor	SS	df	MS	F
(1) Acetic acid concentration (L)	120.4486	1	120.4486	142.2949
Acetic acid concentration (Q)	7.8563	1	7.8563	9.2813
(2) Microwave power (L)	0.0527	1	0.0527	0.0622
Microwave power (Q)	0.6662	1	0.6662	0.7871
(3) Liquefaction time (L)	0.1039	1	0.1039	0.1227
Liquefaction time (Q)	6.1057	1	6.1057	7.2131
IL by 2L	0.0043	1	0.0043	0.0050
1L by 3L	0.1516	1	0.1516	0.1791
2L by 3L	0.4228	1	0.4228	0.4995
Error	5.0788	6	0.8465	
Total	143.6834	15		160.4449

Table 4. Predictive value of optimum maltodextrin dextrose equivalent at critical values of acetic acid concentration, microwave power, and liquefaction time

Factor	Minimum value	Critical value	Maximum value
Acetic acid concentration (%)	6.9546	16.8798	17.0454
Microwave power (watt)	231.8207	390.2327	568.1793
Liquefaction time (min)	28.1821	47.0546	61.8179
Approximate value of dextrose equivalent		17.7632	
Validation value of dextrose equivalent		17.3401	

Parameter optimization for partial hydrolysis of sorghum starch using acetic acid and microwave assistance on acetic acid concentration, microwave power, and liquefaction time was carried out through the determination of critical values shown in Table 3. The critical value obtained for optimizing DE through RSM analysis showed a saddlepoint-shaped distribution, with an estimated DE value for maltodextrin produced

of 17.76328. This optimal value would be achieved at acetic acid concentration of 16.87%, microwave power of 390.2327 watts, and liquefaction time of 47.0546 minutes. Based on the critical value experiment, DE obtained was 17.3401 ± 0.0838, which was close to the predicted value of RSM.

CONCLUSION

In conclusion, this study investigated partial hydrolysis treatment of sorghum starch using acetic acid as a catalyst with microwave assistance. The results showed that the highest DE value of 18.92 was obtained from an acid concentration of 17.04%, microwave power of 400 watts, and liquefaction time of 45 minutes. Meanwhile, the lowest DE value of 7.51 was obtained from an acid concentration of 6,95%, microwave power of 400 watts, and liquefaction time of 45 minutes. This showed that acetic acid concentration and liquefaction time on DE value were directly proportional. Based on the critical value experiment, DE obtained was 17.3401 ± 0.0838, which was close to the predicted value of RSM.

CONFLICT OF INTEREST

There is no conflict of interest.

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