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Optimization of Microwave-Assisted Extraction (MAE) Time and Material to Solvent Ratio of *Gembili* (*Dioscorea esculenta*) Water-Soluble Polysaccharides (WSP)

Optimasi Lama Waktu Proses Microwave Assisted Extraction *dan Rasio Bahan terhadap Pelarut pada Ekstraksi Polisakarida Larut Air Gembili (Dioscorea esculenta)*

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Abstract

Gembili (*Dioscorea esculenta*) is a tuber-producing plant containing 14.63% inulin, a Water-Soluble Polysaccharide (WSP). This study aims to determine the optimum point of the material to solvent ratio and Microwave-Assisted Extraction (MAE) time needed to extract *gembili* WSP, and determine the WSP's characteristics produced. The optimization process uses the Response Surface Methodology-Central Composite Design (RSM-CCD) with Design Expert 7.0 software. The material to solvent ratio's minimum and maximum points are 1:25 g/L and 1:45 g/L, while the minimum and maximum points of extraction time are 20 minutes and 40 minutes. This study observed 13 experimental combinations and responses from *gembili* WSP yield, WSP powder inulin levels, and WSP solubility. The results showed that the optimum conditions of material to solvent were at a 1:33.81 g/mL ratio, and the extraction time was 29.26 minutes. This optimum condition resulted in 32.42% WSP yield, 40.8% inulin content in WSP powder, 26.98% WSP solubility, 12.24% WSP water content, and 49.3 cp WSP viscosity. These results indicate that extraction using a microwave can increase WSP's rich inulin yield from *gembili* tubers.
Keywords: *gembili*, inulin, microwave, optimization, Water-Soluble Polysaccharides

Abstrak

Gembili (Dioscorea esculenta) merupakan tanaman penghasil umbi yang mengandung 14,63% inulin, yang merupakan Polisakarida Larut Air (PLA). Tujuan penelitian ini untuk mengetahui titik optimum rasio bahan terhadap pelarut dan lama waktu ekstraksi PLA ubi gembili yang dibutuhkan menggunakan Microwave-Assisted Extraction (MAE) *serta untuk mengetahui karakteristik PLA umbi gembili yang dihasilkan. Proses optimasi menggunakan* Respon Surface Methodology-Central Composit Design *(RSM-CCD) dengan bantuan software Design Expert 7.0. Titik minimum dan maksimum rasio bahan:pelarut adalah 1:25 g/L dan 1:45 g/L, sedangkan titik minimum dan maksimum lama waktu ekstraksi adalah 20 menit dan 40 menit. Penelitian ini mengamati 13 kombinasi percobaan dan respon, yaitu rendemen PLA, kadar inulin pada bubuk PLA, dan kelarutan PLA. Hasil penelitian menunjukkan bahwa kondisi optimum bahan:pelarut terdapat pada rasio 1:33,81 g/mL dan lama waktu ekstraksi adalah 29,26 menit. Kondisi optimum ini menghasilkan 32,42% rendemen PLA, 40,8% kadar inulin pada bubuk PLA, 26,98% kelarutan PLA, 12,24% kadar air PLA, dan 49.3 cp viskositas PLA. Hasil tersebut menunjukkan bahwa ekstraksi menggunakan microwave berpotensi meningkatkan rendemen PLA kaya inulin dari umbi gembili.*
Kata kunci: *gembili, inulin, microwave, optimasi, Polisakarida Larut Air*

INTRODUCTION

Gembili (*Dioscorea esculenta*) is a Dioscoreaceae family that grows naturally in Indonesia. These plants produce tubers used as an alternative source of carbohydrates to replace rice by the community. *Gembili* has low economic value, so they are less desirable to cultivate as the community's main crops. *Gembili* contains Water-Soluble Polysaccharide (WSP), which is rich in inulin compounds (14.63%) (Winarti, Harmayani, & Nurismanto, 2011). Inulin is a type of WSP consisting of D-fructose, linked by β (2-1) glucosidic bonds and one D-glucose molecule as its terminal, which is connected to the fructose chain by α (2-1) bonds. The polymerization degree of inulin generally ranges from 2 to 60. Inulin is white with a neutral taste (Yang et al., 2015). Statistics Indonesia stated that foreign trade imports of inulin until February 2014 reached 1,231,000 kg or valued at $3,769,901, while foreign trade statistics on imports of inulin until December 2016 reached 7,193,857 kg or valued at $16,536,044 (Statistics Indonesia, 2014; Statistics Indonesia, 2016). Therefore, Indonesia needs to develop research on inulin extraction to find sources of inulin from local plants.

Inulin is located in the vacuole cell plants (Van Laere & Van Den Ende, 2002); therefore extraction process is needed to obtain these compounds. Extraction of inulin compounds from *gembili* tubers generally uses conventional methods, dissolving inulin at a temperature of 80-90 °C for 1 hour (Istinah, 2010). This method is inefficient because the extraction takes a long time, so a new approach is needed. Microwave-Assisted Extraction (MAE) is an extraction method using microwave energy. Extraction using microwave energy will cause dipole rotation in the molecule, resulting in rapid heating of the sample and solvent so that the extraction time is shorter and more efficient. According to Xiao et al. (2013), extraction using a microwave will increase the yield of inulin from the *Helianthus tuberosus* to 12.2%, while extraction using conventional methods only produces a 10.8% yield. The microwave extraction time only takes 6 minutes, while the extraction using conventional methods takes up to 100 minutes.

The extraction time factor is crucial because it affects the microwave energy quantity needed and extraction success rate. This energy is emitted to the material and water as a solvent, resulting in an accumulation of heat energy, increasing the material cells' pressure. The cell material will swell and break then inulin can quickly get out of the matrix cell into the solvent (Maran et al., 2015). The longer extraction time will cause partial hyperthermia due to the internal microwave heating. The inulin fraction will be degraded into monosaccharides, reducing the inulin yield (Tewari et al., 2015). The material ratio to the solvent also needs to be observed. The more available water molecules will support the dipole reaction, which produces heat energy in the cell. The heat energy then accumulates so that the pressure from inside the cell increases, damaging the cell material. This will make the cell contents, including inulin, easily dissolve into the solvent (Guo et al., 2001). However, increasing the solvent ratio too high will decrease the material cell dipole reaction, making it difficult for inulin to get out of the material matrix cell because it can not break down (Li et al., 2015). This solvent's increase resulted in a less optimal extraction process.

Optimizing extraction time and material to solvent ratio is needed for their role in the WSP extraction process using MAE. Response Surface Methodology (RSM) is a collection of mathematical methods and statistical techniques that have been tested and successfully used to optimize the factors that influence the MAE process. The advantage of using the RSM method is that it can reduce the number of experimental units even though many factors will affect the research response (Tewari et al., 2015). This study aims to determine the optimum point of extraction time and material to solvent ratio of *gembili* WSP using MAE, then select the characteristics of WSP produced.

METHODS

Tools and Materials

The tools used for WSP extraction of *gembili* tubers in this study are knives, basins, spatulas, spoons, measuring cups, blenders, mortars, droppings, filter cloth, thermometers, digital scales, cabinet dryers, microwave ovens (Samsung), and freezers. The tools used for inulin characterization are glassware, electric stove, vortex, laminar airflow, desiccator, analytical scale, oven, UV-VIS spectrophotometer, viscosimeter (Elcometer), and scanning electron microscope (Hitachi TM3000). The raw material used for WSP extraction are water, *gembili* tubers (*Dioscorea esculenta*) obtained from Kricak Karangan Kidul Village, Bajeng District, Gresik Regency, East Java, Indonesia. The tubers are harvested at 8-9 months with a tuber weight of 50-120 grams. The chemicals used for WSP characterization are distilled water (Hydrobat), vanillin, H_2SO_4 (Merck), and inulin (Xi'an Lyphar Co., Ltd, China).

Preliminary Experiment

The preliminary experiment aims to determine the water content and inulin content of fresh *gembili* tubers' characteristics and determine the experiment center point based on the highest WSP yields produced. Table 1 shows the basis for selecting the center point of the main research. Thirty minutes MAE time and 1:35 g/mL material to solvent ratio are chosen for the main research design using Response Surface Methodology-Central Composite Design (RSM-CCD) in the Design Expert 7.0.

Main Experiment

The main experiment aims to extract *gembili* WSP based on an experimental design using the RSM-CCD found in the Design Expert 7.0 application. The center point in this study is the 30 minutes extraction time and material to solvent ratio of 1:35 g/mL. The minimum to maximum material to solvent ratio factor is 1:25 g/mL to 1:45 g/mL. While the duration for the minimum to maximum extraction is 20 minutes to 40 minutes. Table 2 shows the experimental research design. The responses measured were WSP yield, inulin content, and solubility.

Gembili with the same color and size is selected so that the sample is uniform. *Gembili* then washed, peeled and weighed according to each treatment. The *gembili* size is reduced using a blender with water as a solvent for each treatment until it becomes 1 L volume of *gembili* pulp. The *gembili* pulp extraction time of each treatment using a 1000 W microwave. A filter cloth was used to filter the extracted *gembili* pulp to obtain two products: *gembili* filtrate and *gembili* dregs. The *gembili* filtrate deposition process is at 21 °C for 20 hours, and then the thawing process is for 1 hour. *Gembili* WSP is separated from water solvent by filtering. The WSP dried for eight hours using a cabinet dryer at 60 °C. A blender and a sieve were used to refine the dried WSP powder.

Data Analysis

WSP powder analysis was carried out based on research responses; yield (Pontes et al., 2016), inulin content using vanillin sulfur method (Levine & Becker, 1959), solubility (Winarti et al., 2013; Kusumayanti, Handayani, & Santosa, 2015), viscosity (Winarti et al., 2013), moisture content (Association of Official Analytical of Chemist, 2015), and brightness (Delgado & Bañón, 2018). The research results data were analyzed using the RSM-CCD method and software Design Expert 7.0 to predict the optimum value of extraction time and material to the solvent ratio in the WSP extraction process using a microwave. Data verification was carried out based on RSM-CCD statistical recommendations from the process optimization results. The significant differ-

Table 1. Preliminary experiment data

Experiment	Extraction Time (Minutes)	Material to Solvent Ratio (g/mL)	Yield (%)
1	20	1:25	29.18
2	25	1:30	30.36
3	30	1:35	32.74
4	35	1:40	26.58
5	40	1:45	25.91

Table 2. Combined research experiments Response Surface Methodology (RSM)-Central Composite Design (CCD)

Run	Code		Factor (X)	
	Extraction Time (X_1) (Minutes)	Material to Solvent Ratio (X_2) (g/mL)	Extraction Time (X_1) (Minutes)	Material to Solvent Ratio (X_2) (g/mL)
1	0.0	0.0	44.14	1:35
2	-1.0	-1.0	20	1:25
3	0.0	0.0	20	1:45
4	1.0	-1.0	30	1:35
5	0.0	0.0	30	1:35
6	0.0	0.0	40	1:45
7	-1.0	1.0	30	1:35
8	0.0	1.4	30	1:49.14
9	1.0	1.0	30	1:35
10	1.4	0.0	30	1:20.86
11	0.0	-1.4	40	1:35
12	0.0	0.0	30	1:35
13	1.4	0.0	15.86	1:35

-1 = Minimum Limit. 0 = Midpoint. +1 = Maximum Limit ence test between *gembili* WSP and standard WSP using paired t-test with Minitab 16 software. The test was carried out on the WSP inulin content characterization, solubility, viscosity, and brightness. WSP structure analyzed using a Scanning Electron Microscope (SEM).

RESULTS AND ANALYSIS

Raw Material Characteristics

Gembili tubers contain Water-Soluble Polysaccharide (WSP) compounds (Herlina et al., 2011). WSP are carbohydrates that can dissolve in water but cannot digest by the human body. The dominant gembili WSP is inulin. This study indicated that the *gembili* has 7.17% inulin content and 72.50% moisture content. These are the average results from 3 replications of *gembili* inulin and water content analysis. Several previous studies have shown different levels of inulin and water content in *gembili*. Bastom (2018) stated that fresh *gembili* contain up to 7.75% water-soluble carbohydrates, with a 72.35% water content. Winarti et al. (2011) stated that *gembili* tubers inulin reached 14.63%, with a water content up to 84.57%. Several factors probably cause this difference; *gembili* harvest age, *gembili* tubers type, *gembili* tubers grown locations, the analyzing tool, and method used.

Optimization of Material to Solvent Ratio and Microwave-Assisted Extraction Time of *Gembili* WSP

Optimization of inulin-rich *gembili* WSP extraction was carried out using RSM-CCD in the Design Expert 7.0. The optimized treatment in this study consists of two factors: MAE time and the material to solvent ratio. The responses optimized were WSP yield, WSP powder inulin content, and WSP solubility in water solvents. WSP extraction optimization aims to obtain optimum WSP yield with good characteristics. The minimum and maximum values of extraction time were 20 minutes and 40 minutes, respectively, while the minimum and maximum values of the material to solvent were 1:25 g/mL and 1:45 g/mL. Table 3 shows the combination of RSM-CCD treatment and response analysis data.

Modeling and Analysis of *Gembili* WSP Yield Response

The best model selection on *gembili* WSP yield response by the Design Expert 7.0 program shows that the quadratic model is suitable for seeing the relationship between extraction time and the material to solvent ratio with the *gembili* WSP yield response. Table 4 shows that the material to solvent ratio variable, extraction time variable, the extraction time squared, and the material to solvent ratio squared significantly affect the *gembili* WSP yield response. The interaction between extraction time and material to solvent ratio variable has no significant effect on *gembili* WSP yield response. The equation obtained based on the analysis of variance results of WSP yield response using the quadratic model is as follows:

$$Y_1 = -38.84 + 1.80X_1 + 2.72X_2 - 2.40\, X_1X_2 - 0.03X_1^2 - 0.04\, X_2^2 \quad (1).$$

Table 3. Data on response analysis of *gembili* WSP yield, inulin content, and solubility

Std	Run	Factor		Response		
		Extraction Time (minutes)	**Material to Solvent Ratio (g/mL)**	**WSP Yield (%)**	**WSP Inulin Content (%)**	**WSP Solubility (%)**
6	1	44.1	1:35	24.71 ± 0.27	28.82 ± 0.39	12.75 ± 0.97
1	2	20	1:25	27.25 ± 0.20	31.70 ± 0.08	17.35 ± 0.15
3	3	20	1:45	25.43 ± 0.15	29.27 ± 0.31	14.51 ± 0.21
10	4	30	1:35	33.10 ± 0.04	41.91 ± 0.06	26.57 ± 0.18
9	5	30	1:35	32,08 ± 0.29	41.06 ± 0.33	26.45 ± 1.82
4	6	40	1:45	24.33 ± 0.30	29.94 ± 0.22	13.89 ± 0.53
11	7	30	1:35	32.76 ± 0.09	41.62 ± 0.18	27.52 ± 0.27
8	8	30	1:49.1	22.24 ± 0.07	29.07 ± 0.05	14.57 ± 1.17
12	9	30	1:35	32.71 ± 0.29	40.28 ± 0.24	25.35 ± 0.39
7	10	30	1:20.8	26.31 ± 0.18	32.21 ± 0.18	18.03 ± 0.08
2	11	40	1:25	27.11 ± 0.18	30.42 ± 0.18	15.25 ± 0.24
13	12	30	1:35	32.42 ± 0.15	41.53 ± 0.21	26.64 ± 0.20
5	13	15.8	1:35	27.84 ± 0.33	32.13 ± 0.06	14.62 ± 1.32

Table 4. Analysis of variance on the response to *gembili* WSP yield

Response	Model	Variable	*P-value*	
WSP yield	Quadratic	X_1	0.0205	Significant
		X_2	0.0010	Significant
		X_1X_2	0.4985	Not significant
		X_1^2	0.0001	Significant
		X_2^2	0.0001	Significant

X_1= Extraction Time (Minutes)
X_2= Material to Solvent Ratio (g/mL)

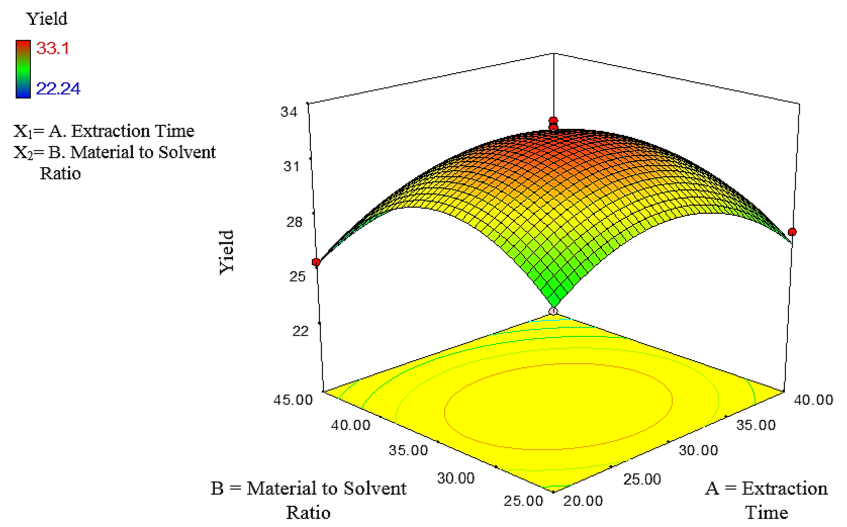


Figure 1. 3D Curve of *Gembili* WSP Yield

Equation (1) can be used to determine the response value of *gembili* WSP yield extracted if the material to solvent ratio and the extraction time is different. This equation shows that the *gembili* WSP yield is high if the extraction time variable (X_1), the material to solvent ratio variable (X_2), and the squared interaction of the extraction time variable with the material to the solvent ratio (X_1X_2) are high. The negative value at the extraction time squared (X_1^2) and the material ratio to solvent squared (X_2^2) indicate a maximum peak point of yield increase.

Figure 1 shows that the optimum point of *gembili* WSP yield increase occurred by 30 minutes of extraction time, resulted in the optimum *gembili* WSP yield of 33.12%. The material cells absorb the microwaves, so the material cells are swelling. The material cell then breaks, makes the cell contents come out quickly into the solvent, increasing the extraction yield (Yang et al., 2010). After passing the extraction time maximum point, the *gembili* WSP yield decreased until the longest extraction time (40 minutes). The heat accumulated during microwave exposure at the extraction time causes the extraction temperature very high. This high temperature causes the degradation of WSP, makes the extraction yield reduced (Maran et al., 2015). The trend of extraction yield decrease when applying a long extraction time also occurred in the extraction of polysaccharides from the *Gentiana scabra Bunge* using the microwave studied by Cheng et al. (2017).

The increase in *gembili* WSP yield was also seen until the optimum point of materials to the solvent ratio (X_2) 1:35, which resulted in 33.12% *gembili* WSP yield. This increase of yield because the water solvent at this ratio can absorb microwave energy efficiently and then triggers the material cells to swell, increasing the surface contact area between the material and the solvent. The target compound easily escapes into the solvent when the cell wall is broken or damaged, increasing extraction yield (Guo et al., 2001). The decrease in the *gembili* WSP yield occurs when the material to solvent ratio increases beyond its optimum point to the highest ratio of material to solvent (1:40). This condition because the excess solvent can reduce or even block the absorption of microwaves by the material. The material cells are not damaged and can prevent the penetration of the target compound into the solvent, which results in reduced *gembili* WSP yield (Li et al., 2010). The *gembili* WSP yield extracted using microwave in this study was higher than the *gembili* WSP yield obtained by conventional extraction methods, 21.06% (Istianah, Mulyani, & Winarti, 2010).

Modeling and Analysis of Inulin Level Response in *Gembili* WSP Powder

The selection of the best model for the inulin levels response by the Design Expert 7.0 program shows that the quadratic model is the best-recommended model to see the relationship between the ratio of materials to solvents and microwave-assisted extraction (MAE) time to inulin levels in the *gembili* WSP powder. Table 5 shows that the variable extraction time, the variable material to solvent ratio, the extraction time variable squared, and the material to solvent ratio variable significantly affect the inulin content of the *gembili* WSP powder. The interaction between extraction time and the ratio of ingredients to solvent has no significant effect on the *gembili* WSP powder inulin levels. The equation obtained based on the results of the analysis of the yield response using the quadratic model is as follows:

$$Y_2 = -63.39 + 3.04\,X_1 + 3.53X_2 + 4.88X_1X_2 - 0.055\,X_1^2 - 0.054\,X_2^2 \quad (2)$$

Equation (2) shows that the levels of inulin (Y) in the *gembili* WSP powder are higher if the extraction time variable (X_1), the material to solvent ratio variable (X_2), the interaction of the extraction time variable with the material to a solvent ratio (X_1X_2) is high. The negative value at the extraction time squared (X_1^2) and the ratio of the material to solvent squared (X_2^2) indicate that the change in Y will form a parabola that opens downward, which means that there is a maximum peak point. Figure 2 shows that the increase in inulin levels in the *gembili* WSP powder occurred until the optimum extraction time (30 minutes), which resulted in the optimum inulin content of 41.62%. Microwaves cause the cell material to break down so that inulin can easily get out of the cell and fuse with the solvent (Li Yang et al., 2010). The dipole reaction by the material molecules causes these molecules to collide with each other to produce heat energy which then raises the temperature in the cell. The cell material then swells and breaks so that the WSP compound

Table 5. Analysis of variance on the response of inulin levels to the *gembili* WSP powder

Response	Model	Variable	*P-value*	
Inulin levels	Quadratic	X_1	0.0443	Significant
		X_2	0.0114	Significant
		X_1X_2	0.2428	Not significant
		X_1^2	0.0001	Significant
		X_2^2	0.0001	Significant

X_1 = Extraction time
X_2 = Material: solvent ratio

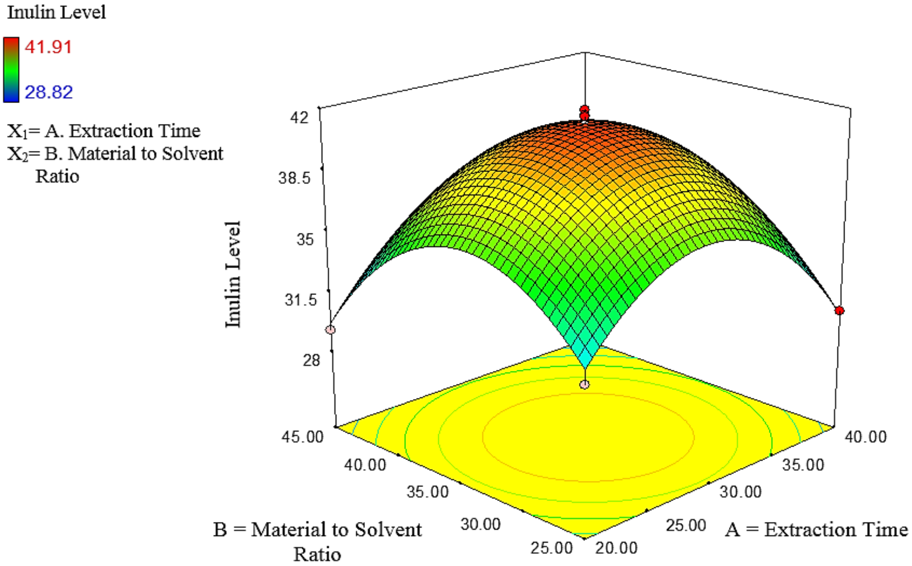


Figure 2. 3D Curve of Inulin Level Response in *Gembili* WSP Powder

quickly into the solvent (Ozcan et al., 2009). The decrease in inulin levels in the *gembili* WSP powder was seen when the increase in extraction time passed its optimum point to the longest extraction time, 40 minutes. Inulin is a carbohydrate polydisperse, and one part of the inulin fraction is very fragile. Microwaves can cause high heating so that the inulin fraction is easily degraded into monosaccharides. This heat reduces the yield of inulin at the time of extraction using a microwave (Tewari et al., 2015).

The inulin content increase in the *gembili* WSP powder was also seen to the optimum point of the material to the solvent ratio (1:35), which resulted in the optimum inulin content of the *gembili* WSP powder (41.62%). The water solvent at this ratio can absorb microwaves efficiently, which triggers an increase in swelling of the material cell and increases the surface contact area between the material cell and the solvent so that the target compound can easily enter the solvent when the material cell breaks (Guo et al., 2001). This benefit makes the levels of inulin in the *gembili* WSP powder increase to their optimum point. The decrease in inulin levels in the *gembili* WSP powder was also seen when the ratio had passed its optimum point to the highest ratio of material to solvent (1:45. An excessive solvent can reduce the absorption of microwaves by the material. The material's cells are not damaged and difficult for inulin compounds to get out into the solvent (Li et al., 2015). It causes a reduction in inulin levels in the WSP powder of *gembili* tubers.

Modeling and Analysis of Variety of *Gembili* WSP Solubility Responses

The quadratic model is the Design Expert 7.0 program choosen to see the relationship between extraction time and material to solvent ratio in the solubility response *gembili* WSP powder. Table 6 shows that the variable extraction time (X_1), the variable material to the solvent ratio (X_2), the extraction time variable squared (X_1^2), the material to solvent ratio variable (X_2^2) have a significant effect on the *gembili* WSP solubility response. In contrast, the interaction variables extraction time with the variable material to the solvent ratio (X_1X_2) had no significant effect on the solubility response. The equation obtained based on the analysis of variance is as follows:

Table 6. Analysis of variance on *gembili* WSP solubility response

Response	Model	Variable	*P-value*	
Solubility	Quadratic	X_1	0.0168	Significant
		X_2	0.0011	Significant
		X_1X_2	0.2626	Not significant
		X_1^2	0.0001	Significant
		X_2^2	0.0001	Significant

X_1 = Extraction time
X_2 = Material to solvent ratio

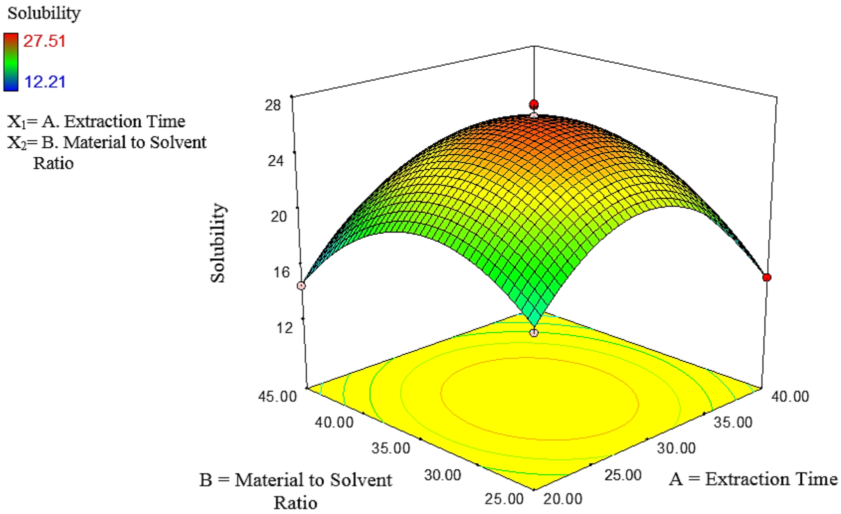


Figure 3. 3D Curve of Solubility Response of *Gembili* WSP

Y_3= -80.28 + 3.65X_1 + 3.18X_2 + 5.025X_1X_2 – 0.065X_1^2 – 0.049X_2^{2}. (3).

Equation (3) shows that the solubility value of inulin (Y) increases if the extraction time variable (X_1), the material to solvent ratio variable (X_2), the interaction between the extraction time variable and the material to solvent ratio variable (X_1X_2) gets higher up to the optimum point then there is a decrease which is indicated by a negative value at X_1^2 and X_2^2

Figure 3 shows that the *gembili* WSP solubility value increases up to the optimum point of extraction time (30 minutes), which results in a WSP solubility value of 27.51%. The inulin content in *gembili* WSP powder also increased during that time until it reached its optimum point, which was 41.91%. The solubility of inulin is influenced by the hydroxyl (OH) groups possessed by inulin compounds so that inulin is polar, which makes it easier for it to dissolve in water solvents. The inulin content of the soluble *gembili* WSP affects the solubility percentage of the *gembili* WSP (Winarti et al., 2011). Figure 3 also shows a decrease in the *gembili* WSP solubility response value when passing the optimum point of extraction time until the longest extraction time (40 minutes), resulting in a *gembili* WSP solubility value of 12.21%. Due to decreased inulin levels during that time, the solubility response value of WSP is getting lower.

The increase in *gembili* WSP solubility response value was also seen until the optimum ratio of material to solvent (1:35), which resulted in *gembili* WSP solubility of 27.51%. The inulin content in the *gembili* WSP powder also increased to reach 41.91%, up to the optimum point for the ratio of the substance to the solvent (1:35). The solubility of inulin compounds in the *gembili* WSP powder caused an increase in the solubility response of the *gembili* WSP. A decrease in the value of the solubility response then occurs when the ratio of the substance to solvent passes the optimum point to the highest ratio of material to solvent (1:45), which results in a solubility value of 12.21%. The decrease in the solubility response was due to a decrease in inulin levels in the *gembili* WSP. There will be fewer inulin compounds that dissolve, which causes a low solubility response value of the *gembili* WSP.

Determination of the Optimum Point and Research Verification

Determining the optimum point in this study was based on the in-range criteria for the extraction factor and the maximization criteria in the research response using the Design Expert 7.0 program. Research verification was done by extracting the *gembili* WSP at the value of the extraction time factor and the material to solvent ratio predicted by the Design Expert 7.0 program. The response analysis was carried out on the *gembili* WSP yield, insulin levels, and solubility. The real difference test by the response value was predicted by the Design Expert 7.0 program using the paired t-test using Minitab software with α = 0.05.

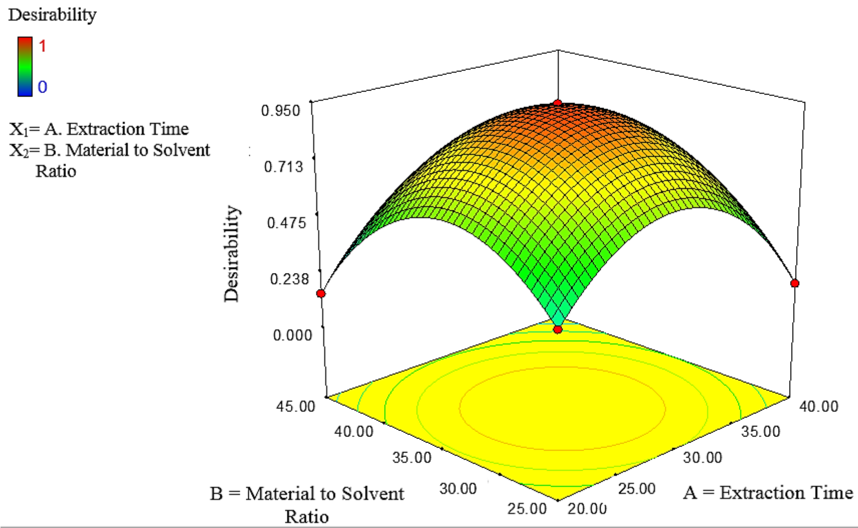


Figure 4. 3D Curve of the Design Expert Program Prediction 7.0 Optimal Point Results

Table 7. Data for optimum point solution and research verification

	Extraction Time (Minutes)	Material to Solvent Ratio (g/mL)	WSP Yield (%)	WSP Inulin Levels (%)	WSP Solubility (%)
Predictions	29.26	1: 33.79	32.74	41.33	26.59
Verification	29.26	1: 33.81	32.42 ± 0.21	40.8 ± 0.28	26.98 ± 0.34
p-value	-	-	0.403	0.082	0.272

Table 8. Physical and chemical characteristics of *gembili* WSP

Parameter	*Gembili* WSP	Standard WSP (Inulin)	p-value (α=0.05)
Inulin Level (%)	40.8 ± 0.28	97.00 ± 0.3	0.000
Solubility (%)	26.98 ± 0.34	99.64 ± 0.37	0.000
Brightness (L)	67.6 ± 0.34	91.21 ± 0.12	0.000
Viscosity 10% (cp) 90o C	49.3 ± 0.57	4.70 ± 0.58	0.000

The extraction time, material to the solvent ratio, yield response, response to inulin levels, and the solubility response *gembili* WSP recommended by the Design Expert 7.0 program have a desirability value of 0.9 (Figure 4). It indicates that the program's ability to provide an optimum response follows the character specified. The response data for the *gembili* WSP yield, the response of inulin levels, and the solubility response from the Design Expert 7.0 program, the results of verification, and the paired t-test results are shown in Table 7.

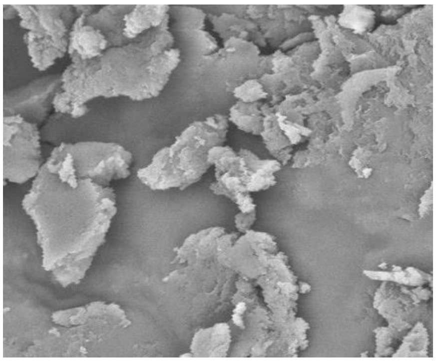
The paired t-test using Minitab software shows that the p-value in all research responses is more significant than α = 0.05. It means that there is no difference between the formula recommended by the Design-Expert 7.0 program and the results of research verification. It can be concluded that the extraction time and the ratio of materials to solvents recommended by the Design-Expert 7.0 program are correct.

Chemical and Physical Characteristics of *Gembili* WSP Optimization Results

The optimization *gembili* WSP result with an extraction time of 29.26 minutes and a ratio of ingredients to solvent 1: 33.81 resulted in a *gembili* WSP yield of 32.42%, inulin levels of 40.08%, and solubility of 26.98%. The *gembili* WSP was then compared with the standard WSP. The comparison includes the value of inulin content, solubility value, brightness value, and viscosity value at a concentration of 10%, a temperature of 90 °C. A comparison of chemical and physical characteristics of the optimized WSP and standard WSP is shown in Table 8. The microstructure observation of the WSP of *gembili* tubers was also carried out using Scanning Electron Microscope (SEM) to see the starch compounds extracted as impurities in the WSP of *gembili* tubers.

Table 8 shows that the inulin content in the optimized *gembili* WSP was 40.8%, while the inulin content in the standard WSP powder reached 97%. The paired t-test using Minitab software showed that *gembili* WSP inulin levels and standard WSP were significantly different at α = 0.05. The inulin content was lower than the results obtained by Winarti et al. (2011), extracting the *gembili* inulin with a purity of 73.58%. The low inulin content in the optimized *gembili* WSP is probably because many other compounds such as glucomannan, protein, and starch are extracted. Glucomannan is a type of other WSP found in *gembili*. The glucomannan content in the *gembili* WSP is 39.49% (Herlina, 2012). *Gembili* also contains protein of 5.74% (wb) and amylose of 3.84% (wb) (Herlina et al., 2011). The two compounds were also extracted as unwanted impurities in the *gembili* WSP extraction.

Table 8 shows that the *gembili* WSP solubility is 26.98%, while standard WSP is 99.64%. The results of statistical tests using paired t-test showed that the *gembili* WSP solubility was significantly different at the level of α = 0.05 with standard WSP powder. The low *gembili* WSP solubility probably due to cabinet dryers in the *gembili* WSP drying process. According to Panchev et al. (2011), drying inulin can cause inulin to be semi-crystalline. The semi-crystalline nature will form the characteristics of the inulin texture, which is hard and difficult to interact with other materials (Winarti et al., 2013). The commercial inulin production process generally uses spray dryer drying, producing amorphous properties and is very soluble in water solvents.


(a) *Gembili* WSP

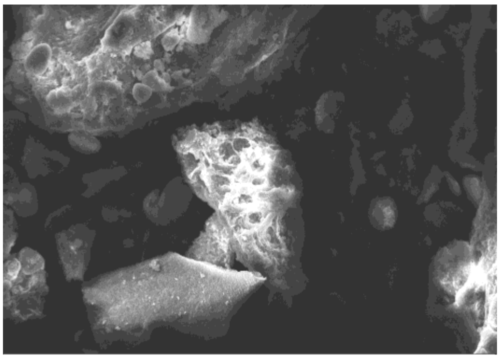

(b) *Uwi* WSP
(An-nuha & Sunarti, 2018)

Figure 5. Observation of WSP Microstructure

Measuring the viscosity of *gembili* WSP and commercial WSP at a temperature of 90 °C with a concentration of 10% showed that the *gembili* WSP viscosity was 49.3 cp, while the viscosity of standard WSP powder was 4.7 cp. The paired t-test showed that the viscosity of the optimized *gembili* inulin powder was significantly different from the viscosity of standard WSP powder. This different because *gembili* WSP may also contain glucomannan and dioscorin compounds. According to Myoda et al. (2006), the viscosity of WSP is influenced by the content of manan and protein dissolved in it. Yeh, Chan, & Chuang (2009) also stated that WSP has high hydration power, which causes WSP to absorb more water to increase the *gembili* WSP's viscosity.

The brightness (L) of the optimized *gembili* WSP was 67.6, while the brightness of the standard WSP powder was 91.2. The paired t-test showed that the brightness level of the optimized *gembili* inulin powder was significantly different from the brightness level of the commercial inulin powder. This brightness difference is probably due to the impurity in the *gembili* WSP powder due to the optimization of protein compounds that dissolve in the extraction process of *gembili* inulin. According to Prabowo, Estiasih, & Purwantiningrum (2014), the *gembili* contains dioscorin compounds as storage proteins in the Dioscorea family that dissolve quickly in water solvents. Protein compounds and reducing sugars in *gembili* WSP may cause Maillard reactions which form a brownish color during the optimization of the WSP powder drying process (temperature 50 °C for 10 hours) by cabinet dryer so that the brightness of the WSP powder decreases.

Microstructure observation using SEM aims to see the *gembili* tubers WSP microstructure extracted using a microwave. The results of microstructure observations on WSP *gembili* tubers (Figure 5a) show that the *gembili* WSP shape extracted using a microwave is similar to the uwi tubers WSP microstructure extracted at 50 °C by An-nuha & Sunarti (2018). According to Wang, Xiao, & Li (2008) microwaves will be absorbed by the material and water cells causing a dipole reaction resulting in a rapid increase in temperature. This temperature increase causes the cell wall to swell and burst so that the WSP compound will be released from the cell into the solvent.

CONCLUSIONS

The Design Expert 7.0 program prediction shows that 29.26 minutes extraction time and 1: 33.81 ingredients to solvent ratio is the optimum formula for extracting *gembili* tubers WSP using a microwave. The verification results showed 32.42% WSP yield, 40.8% WSP powder inulin levels, and 26.98% WSP solubility. WSP *gembili* tuber powder has physical characteristics of 49.3 cp viscosity at a concentration of 10% with a temperature of 90 °C.

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