



Response surface methodology in the optimization of walur (*Amorphophallus paeoniifolius* var. *Sylvestris*) starch pregelatinization process

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KEYWORDS

Pregelatinization
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ABSTRACT

This study aimed to determine the effect of temperature and time of the pregelatinization process on the physical and functional properties of pregelatinized and native walur starch using the central composite design method of response surface methodology (CCD-RSM). Several analyses, including rapid visco analyzer (RVA), scanning electron microscopy (SEM), and X-ray diffraction (XRD), were used to characterize an optimum pregelatinized walur starch (PWS). The optimum conditions for producing PWS were at 87.51°C and 9.71 minutes. The experimental verification data, repeated three times, were not significantly different ($P > 0.05$) from the prediction optimization data generated by the Design Expert Software 7.1.5 Trial Version, which produces PWS with 19.56 ± 0.68 % swelling, 9.87 ± 0.18 % solubility and 835.62 ± 0.84 % water holding capacity (WHC). The result from RVA analysis showed that the pregelatinization process of walur starch increases the peak, final and setback viscosity, peak time, and pasting temperatures but decreases breakdown viscosity. Native walur starch (NWS) had a more crystalline form than PWS on XRD analysis. The SEM analysis revealed that NWS had smooth surface granules compared to PWS granules.

Introduction

Amorphophallus paeoniifolius var. *Sylvestris* is common species in Java. The species have two common names: 'walur' and 'suweg' (Yuzammi et al., 2017). 'Walur' has rough petioles and grows wild (weedy). In contrast, 'suweg' has an almost smooth petiole, is cultivated, and has economic value for rural people as a staple food (Mutaqin et al., 2020). Walur tuber has no economic value than other tubers in the *Amorphophallus* family and is non-edible due to its oxalate content. The tubers are very stingy and toxic for human consumption. In some cases, 'walur' chips have been used to adulterate yellow konjac or 'porang' (*Amorphophallus muelleri*) chips. Porang chips are more expensive than other tubers in the *Amorphophallus* family. Purnomo et al. (2011) reported that the total starch content of walur was 65.72% on a dry basis, and it can be used for cookies and noodle making. To the best of our knowledge, a limited number of papers reported on walur starch, particularly pregelatinized starch.

Starch has been known as one of the ingredients in many food and non-food industries

(Oladebeye et al., 2013). Starch for food purposes is chosen because of its swelling power (SP), solubility (S), and water holding capacity (WHC). It is rarely used in industry in its native form because of its solubility restriction in water and other common organic solvents, thermal decomposition, low shear stress resistance, and high tendency on retrogradation and syneresis (Alcázar-Alay & Meireles, 2015; Neelam et al., 2012). Modifying the starch can overcome these starch limitations (Ulfa et al., 2021). Starch modification techniques can be classified into four categories: chemical, enzymatic, physical, and genetic modifications (Neelam et al., 2012).

Modified starches are one of commercial interest for use in food industries. Physical modification techniques are facile and often inexpensive compared to chemical modification. Heat-moisture treatment is one modification method to disrupt the granule starch structure and induce the required functional properties. Pregelatinization is effectively improve starch functional characteristics by using high temperatures to gelatinize the starch before drying

(Abbas et al., 2010). The research and exploration of new starch resources have always been a hotspot in starch research.

A group of mathematical and statistical techniques known as “Response Surface Methodology” or RSM can describe the relationship between several independent variables and one or more responses, useful for developing, improving, and optimizing the process. It also has essential applications in the design, development, formulation of new products, and the improvement of existing product designs (Box and Wilson, 1951; Khare et al., 2015; Myers et al., 2016; Widjanarko et al., 2018).

Optimization is a method for determining the best solution in terms of specific quality criteria (such as process efficiency) and improving the performance of the designed system or process. This method could be divided into six steps: (1) choosing independent variables and potential responses, (2) choosing experimental design strategy, (3) conducting experiments and obtaining results, (4) fitting the model equation to experimental data, (5) obtaining response graphs and verifying the model (ANOVA), (6) determination of optimal conditions (Witek-Krowiak et al., 2014).

The application of RSM in the starch modification process has been reported in the optimization of sweet potato starch pregelatinization process, potato and maize starch blends, and hydrothermally modification process of elephant foot yam starch, which can give the optimum condition process and responses (Ulfa et al., 2021; Barua et al., 2021; Kankate et al., 2020).

This work aimed to investigate the combined effect of temperature and time on the changes in swelling power, solubility, and WHC of pregelatinized walur starch using RSM. To date, this is the first report of the pregelatinized walur starch.

Materials and Methods

Materials

Walur tubers (*Amorphophallus paeoniifolius* var. *Sylvestris*) were collected from local farmers in Madiun Regency, Indonesia, weighing 600-800 g and 12 - 15 cm in diameter. Walur tubers collected from a local farmer are approximately more than two years old of the maturity stage. The starches were extracted from the fresh tubers using established procedures (Purnomo et al., 2011) at our laboratory.

Experimental design and statistical analysis

Central composite design (CCD) method was chosen in the Design Expert 7.1.5 trial program (State-Ease, Inc) with two independent factors, and those are temperature (°C) (X_1) and time (minute) (X_2) (Table 1). The range for each independent factor was based on preliminary experiment results, and three responses: swelling, solubility, and WHC, were chosen as dependent variables. Thirteen experimental points as the total treatment combinations, including five centre points, four factorial points, and four axial points. Multiple regressions analyzed all data in CCD to fit into the empirical second-order polynomial model. (Eq. 1)

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i=1}^k \sum_{j=1}^k \beta_{i,j} X_i X_j + \varepsilon \dots\dots\dots (1)$$

Note:

β_0 = Coefficient of regression in the intercept

β_i = linear

β_{ii} = quadratic

$\beta_{i,j}$ = interaction terms

X_i ; X_{ii} ; $X_{i,j}$ are the independent variables;

Y are responses.

ε is a random error component.

Native walur starch (NWS) preparation

An NWS was prepared using established methods with slight modifications (Purnomo et al., 2011). Tubers were washed, peeled, and trimmed to remove defective parts. The tubers were sliced and soaked in 0.2N HCl solution for 30 minutes and then rinsed thoroughly. After that, the tubers were soaked in 1% sodium bicarbonate (NaHCO_3) solution for 5 minutes and rinsed. The tubers were disintegrated using a Panasonic blender MX-V310 with additional distilled water. A filter cloth was used to screen the yield, and the retained solids were rinsed three times on the filter using distilled water. The filtrate was allowed to stand overnight to collect the precipitate and the supernatant was discarded. The resuspension and sedimentation processes were repeated several times until white starch was obtained. The collected starch was put in a cabinet dryer at 50°C for 8 hours. Dried walur starch was ground using a pestle and a mortar, and the product was passed through an 80-mesh sieve to collect the fine starch powder.

Pregelatinized walur starch (PWS) preparation

The method described by Anggraini et al. (2016) was slightly modified in the starch powder ratio and drying temperature to produce pregelatinized

walur starch (PWS). An aqueous starch was made with starch powder and distilled water with a ratio of 1:20, and heated in a water bath with constant stirring. The resulting paste mixture was taken into a tray and dried in a cabinet dryer at 40°C to get a PWS sample. The temperature and heating time used in this experiment runs were according to the specified in Table 1 and Table 2.

Percent solubility and swelling

The percent solubility and swelling properties were determined using established methods (Odeku et al., 2008). 1% w/w starch suspensions were produced and boiled for 30 minutes in a shaker water bath at 85 ± 1 °C with shaking every 5 minutes and let to cool at room temperature before centrifuged at 3000g for 15 minutes. To obtain S, the supernatant was discharged and dried in an oven at 105 °C for 2 hours. The residue was weighed and calculated as SP. The % solubility and % swelling were assessed by applying Eq. 2 and 3 (Kankate et al., 2020).

$$\% \text{Solubility} = \left(\frac{W_{su}}{W_i} \right) \times 100 \dots\dots\dots(2)$$

$$\% \text{Swelling} = \left(\frac{W_{su}}{W_i \times (100 - \% \text{Solubility})} \right) \times 100 \dots\dots(3)$$

Where, Wsu is the residue's weight, Wi is the initial weight of dried starch, and Wss is the wet sediment starch's weight.

Water holding capacity

An aqueous suspension of 1% (w/v) starch was stirred for 60 min. Furthermore, all the PWS and NWS were centrifuged for 20 minutes at 3500g. The transparent supernatant was removed, the remaining wet part was put into a weigh cup, and the weighed was recorded. % WHC was computed by using Eq. 4 (Kankate et al., 2020).

$$\% \text{WHC} = \left(\frac{W_s}{W} \right) \times 100 \dots\dots\dots(4)$$

Where, Ws is the wet residue's weight, and W is the starch's weight on a dry basis.

Pasting Properties

Profiles of paste starch of PWS and NWS were analyzed using Rapid Visco Analyzer (RVA Tecmaster Newport).

X-ray Diffraction (XRD)

PWS and NWS powders were assessed by deploying the X-ray diffraction (XRD) instrument (PANalytical X'Pert 3 Powder). Those powders firmly filled in a desk glass (a sample holder) were exposed to X-ray beams at 40kV and 30 mA. The canning region of the 2θ angle was set from 5° to 80°. The run time was 10 minutes for each sample.

Table 1. Independent variables and factor levels in the central composite design

Independent Variables	Symbol	Factor Level				
		-1.414	-1	0	+1	1.414
Temperature (°C)	X ₁	80.43	82.50	87.50	92.50	94.57
Time (minute)	X ₂	2.93	5	10	15	17.07

Table 2. Physical properties obtained for each experimental running of central composite design (CCD)

Std	Run	Coded Variable		Actual Variable		Responses		
		X ₁	X ₂	Temperature (°C)	Time (minute)	Swelling (%)	Solubility (%)	WHC (%)
1	7	-1.00	-1.00	82.50	5.00	15.90	9.92	728.16
2	10	1.00	-1.00	92.50	5.00	17.50	7.74	745.37
3	13	-1.00	1.00	82.50	15.00	16.60	9.23	798.20
4	11	1.00	1.00	92.50	15.00	17.50	7.64	805.10
5	5	-1.41	0.00	80.43	10.00	15.8	8.94	725.37
6	12	1.41	0.00	94.57	10.00	16.81	6.16	790.21
7	2	0.00	-1.41	87.50	2.93	20.45	9.32	850.89
8	8	0.00	1.41	87.50	17.07	17.06	8.14	856.66
9	6	0.00	0.00	87.50	10.00	21.00	10.10	850.00
10	4	0.00	0.00	87.50	10.00	20.19	9.61	806.96
11	9	0.00	0.00	87.50	10.00	18.00	10.38	843.92
12	3	0.00	0.00	87.50	10.00	19.74	10.90	850.55
13	1	0.00	0.00	87.50	10.00	21.60	9.60	833.20

Scanning electron microscopy (SEM)

PWS and NWS powders were observed using scanning electron microscopy (SEM) (FEI Quanta FEG 650 FE-SEM type) at 5 keV accelerating voltage. The topography and average value of the particle diameter of both samples were recorded.

Results and Discussion

Model fitting from RSM

The effect of pregelatinization temperature and heating time on swelling, solubility, and WHC are indicated in Table 2. The influence of the independent and dependent variables was fitted to the second-order model equation, and the goodness of fit was evaluated. The analyses of variance (ANOVA) were used to obtain the F_{value} model, and the significance of the linear, interaction, and quadratic effects of independent variables on the dependent variables showed by the lack of fit (Table 3). The lack of fit test indicates that a model failed to represent data in the experimental domain, where points were excluded from the regression (Varnalis et al., 2004). The F_{value} (model) of the quadratic effect of temperature on responses was significant, with not significant lack of fit (Table 3). Non-significant lack of fit is good, and we want the model to fit. The same results were claimed by Ulfa et al. (2021), where the lack of fit of the

independent variables on pregelatinized sweet potato starch dependent variables was insignificant. At the same time, the F_{value} model of the quadratic model was significantly different. R^2 or the total determination coefficient is a variation in responses, namely swelling, solubility, and WHC, rather than random error. R^2 represents the model's fitting quality, the high R^2 value showed that the regression model was well fitted to the data and was suggested as a good fit model (Askari et al., 2021; Roudi et al., 2021). The results showed that the models for all the response variables were adequate, with satisfactory levels of R^2 of more than 70%. It was reported that R^2 for all responses of elephant foot yam starch modified using a hydrothermal process was affected by the independent variables by more than 90% (Barua et al., 2021), and the models were adequate. The regression coefficients are shown in Table 3.

Effect of temperature and heating time

a. Swelling

The swelling power of pregelatinized walur starch (PWS) varied from 15.80% to 21.60% (Table 2). Analysis of regression appeared that swelling PWS was significant ($P < 0.05$) and dependent on heating temperature (Table 3).

Table 3. Regression coefficients and significance of the predicted second-order polynomial models for swelling, solubility, and WHC of walur starch

	df	Regression coefficients		
		Y_1^a	Y_2^b	Y_3^c
Intercept, X_0		20.10	10.12	836.93
Linear				
X1	1	0.49	-0.96**	14.48
X2	1	-0.51	-0.31	17.24
Interaction				
X1X2	1	-0.18	0.15	-2.58
Quadratic				
X1 ²	1	-2.06**	-1.16**	-48.71**
X2 ²	1	-0.84	-0.57*	-0.72
Residual	7			
Lack of Fit	3	0.84	0.64	5.16
Pure Error	4			
Cor Total	12			
Mean		18.32	9.05	806.51
Standard Deviation		1.33	0.50	30.27
R²		0.74	0.91	0.76
Adjusted R²		0.56	0.85	0.60
F-value (model)		4.07*	14.87*	4.54*

* $p < 0.05$; ** $p < 0.01$; ^aswelling (%), ^bsolubility (%), ^cWHC

Equation models for second-order polynomial, which illustrates the correlation between responses and the terms of independent variables (i.e., linear, interactive, and quadratic), are outlined as follows (Eq. 5):

Swelling (%):

$$Y_1 = 20.10 + 0.49X_1 - 0.51X_2 - 0.18X_1X_2 - 2.06X_1^2 - 0.84X_2^2 \text{ (df = 5, R}^2\text{= 0.74) (5)}$$

According to Eq. 5, the temperature had a positive linear effect on PWS swelling. Increasing temperature from 80.43 – 87.5 °C increased swelling from 15.8 – 21.0%. The swelling of starch was found to be a function of temperature. Alam and Hasnain (2009) reported increasing temperature from 20 – 90 °C on modified starch taro (*Colocasia esculenta*) increased percent swelling. The water absorption ability of starch granules may explain why the percentage of swelling increases with temperature. The results in Eq. 5 were in agreement with the finding of Ulfa et al. (2021), where the value of swelling power increases after the pregelatinization process by the increasing temperature given in the process. The ANOVA for the quadratic model showed that percent swelling was significantly (P<0.05) affected by the quadratic temperature and showed a negative impact on percent swelling due to starch granules rupture by increasing temperature up to 90 °C, and starch granules lost their power to absorb water (Figure 1. (A2)). The polynomial equation revealed that the swelling power value that appeared by the cold-water solubility value was affected negatively by independent variables, namely quadratic treatment time (Zhu et al., 2017).

b. Solubility

The solubility of PWS ranged from 6.16 – 10.00 % (Table 2). Results from ANOVA for the quadratic model of solubility indicated that linear temperature, quadratic temperature, and time indicated significantly (P<0.05) influenced the solubility of PWS (Table 3). The interaction of temperature and time had a positive influence on solubility. PWS exhibited higher swelling and solubility in contrast to NWS. The value of swelling and solubility of pregelatinized starch increases with rising temperature and modification time (Singh & Kumar, 2020).

Equation models for second-order polynomials illustrates a correlation between response and the terms of independent variables (i.e., linear, interactive, and quadratic), which are described below (Eq. 6):

Solubility (%):

$$Y_2 = 10.12 - 0.96X_1 - 0.31X_2 + 0.15X_1X_2 - 1.16X_1^2 - 0.57X_2^2 \text{ (df = 5, R}^2\text{= 0.91) (6)}$$

According to Eq. 6, the interaction between temperature and time had a positive effect on the solubility of PWS; it might be due to structural changes within PWS after the pregelatinization process, which might be responsible for the increase in PWS solubility. It was in agreement with the solubility of elephant foot yam starch which increased as the temperature increased during the modification process (Suriya et al., 2019). Modified starches’ higher swelling and solubility values are mainly associated with amylose leaching (Kankate et al., 2020). Response surface 3D plots of solubility affected by temperature and time showed a negative effect in the solubility of PWS due to quadratic temperature and time effect in the solubility of PWS. Quadratic temperature and time revealed that the solubility of PWS was significant (P<0.05) (Table 3), and optimum solubility was exhibited at the temperature of 87.50 °C and time at 10.00 minutes (Figure 1. (B2)). Starch granules' susceptibility to solubility under temperature and time is affected by the amylose concentration and degree of starch's crystallinity (Abedi et al., 2019). The R² for solubility was 0.91 (Table 3), and it was significantly affected (P<0.05) by linear temperature and quadratic temperature and time. This R² value for solubility exceeded 90%, exhibiting high variability data. As a result, the response surface models developed were adequate.

c. Water holding capacity

WHC of PWS ranged from 725.37 – 856.66 % (Table 2). The ANOVA for the linear model of WHC revealed that temperature and time positively affect the WHC of PWS (Table 3). This could be due to increased amylose concentration of pregelatinized starch caused by the degradation of amylopectin molecules during modification starch processes. The increase in WHC of PWS indicates that the crystallinity of pregelatinized starch was disrupted and disorganized, thus increasing the solubility of pregelatinized. The swelling and solubility values increase with the soaring temperature from 30 – 90 °C (Singh and Kumar, 2020), and so does the WHC value of pregelatinized starch.

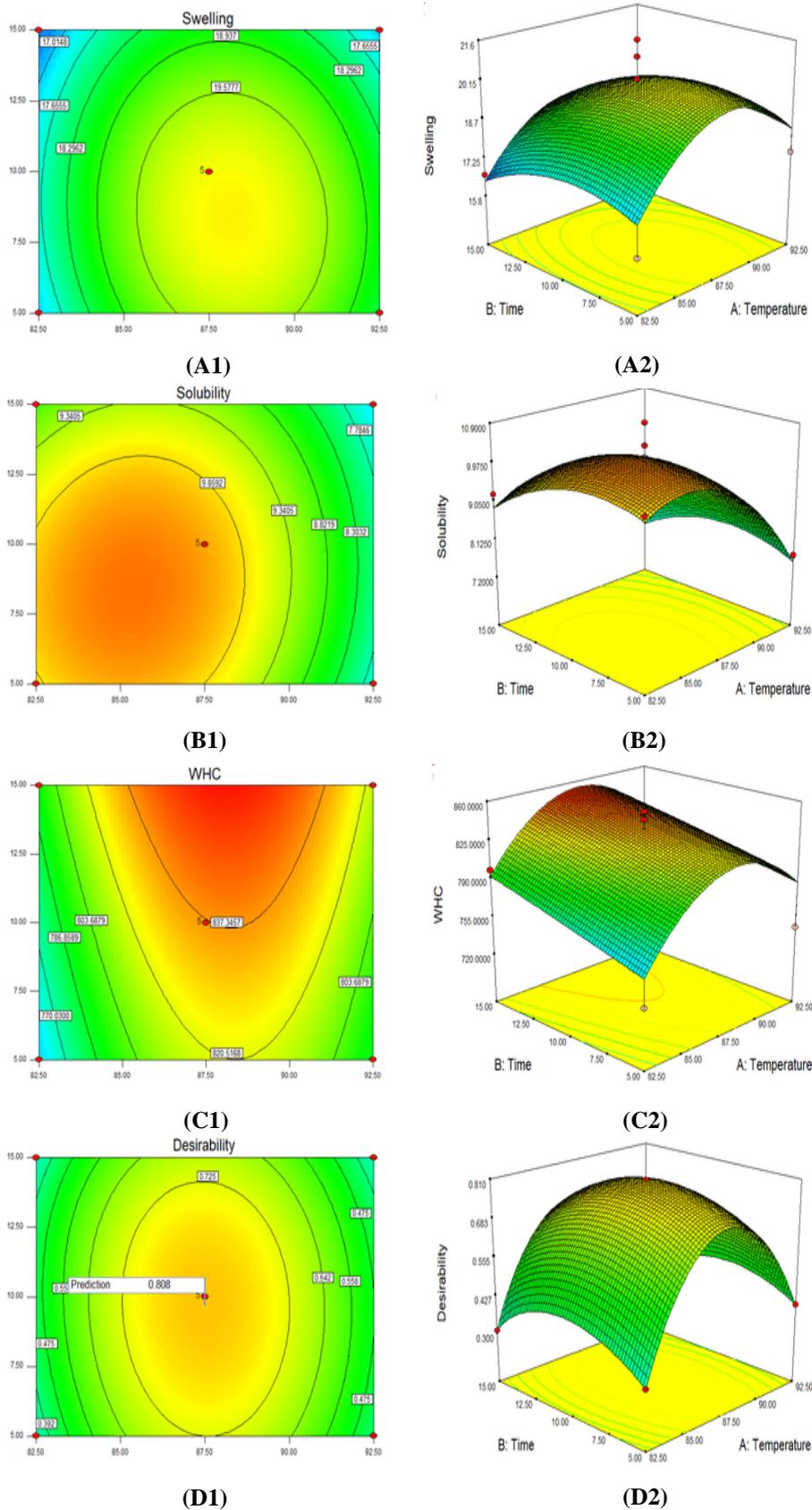


Figure 1. Contour and response surface plots of swelling (A1,A2), Solubility (B1,B2), WHC (C1,C2) of pregelatinized walur starch, and contour and response surface plots of desirability (D1,D2) for predicted optimum result of a quadratic effect of interactions between temperature and time

Equation models for second-order polynomials, which exhibit a correlation between response and the terms of independent variables (i.e., linear, interactive, and quadratic), are outlined below (Eq. 7):

WHC (%):

$$Y_3 = 836.93 + 14.48X_1 + 17.24X_2 - 2.58X_1X_2 - 48.71X_1^2 - 0.72X_2^2 \text{ (df = 5, R}^2\text{= 0.76) (7)}$$

Eq. 7 showed a positive effect of temperature and time on WHC due to starch granules of PWS disrupted and disorganized; as the temperature and heating time increase, so do the swelling power and solubility. Kankate et al. (2020) reported that the WHC value of pregelatinized starch and its mixture increased with increasing temperature. The WHC value of flour refers to its ability to absorb water. WHC is an essential processing parameter and important in bulking. It is expected that moisture uptake will increase as the temperature for gelatinization increases (Niba et al., 2002).

Predicted optimization and verification

The criteria for obtaining the optimization process were set as follows: maximum swelling, maximum solubility, and maximum WHC. The prediction results showed that the optimum condition process was the temperature at 87.51 °C and 9.71 minutes. The prediction produces starch with 20.13 % swelling, 10.13 % solubility, 835.94 % WHC, and 81 % desirability. The obtained desirability means the independent variables affect 81 % of responses and 19 % come from noises. The verification step was conducted using the predicted condition process with three replications. The verification data compared to the predicted data were calculated using Paired T-Test (Minitab version 17). The verification results were pregelatinized starch with 19.56 ± 0.68 % swelling, 9.87 ± 0.18 % solubility, and 835.62 ± 0.84 % WHC. These values were not significantly different (P>0.05) and confirmed that the model was accurate. Similar results were reported by Ghodke et al. (2009).

Pasting properties

NWS and PWS’s pasting properties are shown in Table 4. Pasting starches’ properties differ depending on the granule’s size, the amylose and amylopectin ratio, the rigidity, and the swelling power (Aktaş and Gerçekaslan, 2018). The pasting properties of PWS had similar peak viscosity and pasting temperature, while the through, breakdown, setback, and final viscosity also the peak time compared to NWS were significantly different (P<0.05). Different results were found in the peak viscosity of elephant foot yam starch modified using HMT, the modified starch has lower peak viscosity than its native starch (Suriya et al., 2019). These results indicate that amylose in the starch plays is vital in providing physical modification effects on pasting properties. Heat treatment caused granule swelling and amylose leaching, which then released the amylose and formed the starch matrix due to the aggregation of amylose on the granule’s surface, increasing starch granule rigidity (Gerçekaslan, 2020).

Breakdown viscosity (BV) reflects the measure of the starch’s stability to heat and shear. PWS with a lower BV, which was 16 times lower than BV NWS (Table 4), indicates the cooked starch to disintegration was lower for PWS. Therefore, the paste's stability of PWS was more stable than the NWS paste's stability. Higher peak viscosity and the through viscosity of PWS shows its water absorption capacity, and the swell was increased (Suriya et al., 2017). When the starch granules swell, the system's viscosity increases and the viscosity reaches the peak caused by the highest level of swollen granules amount (BeMiller, 2018). PWS has a higher final viscosity than NWS. The increase of the final viscosity caused by the leached chains of amylopectin and amylose reassociation, resulting in a viscous paste, which might be affected by the high amylose content (Murayama et al., 2014; Suriya et al., 2019). A setback is an in-viscosity occurred during cooling (Gerçekaslan, 2020). The increase in setback viscosity was caused by starch retrogradation, which joined adjacent amylose molecules through molecular hydrogen bonds (Subroto et al., 2019).

Table 4. Pasting properties of pregelatinized and native walur starch

	Pregelatinized walur starch	Native Walur Starch
Peak Viscosity (cP)	4381.00 ^a ± 161.22	4354.00 ^a ± 31.11
Through Viscosity (cP)	4255.00 ^a ± 9.90	2299.00 ^b ± 46.67
Breakdown Viscosity (cP)	126.00 ^b ± 2.83	2055.00 ^a ± 15.56
Final Viscosity (cP)	6199.00 ^a ± 32.53	3012.00 ^b ± 72.12
Setback Viscosity(cP)	1944.00 ^a ± 21.21	713.00 ^b ± 25.46
Peak Time (minute)	6.40 ^a ± 0.01	4.47 ^b ± 0.05
Pasting Temperature (°C)	82.25 ^a ± 0.21	81.08 ^a ± 0.46

SEM results

The pregelatinization process using various temperatures and time creates the disruptive physical nature of granular starch. NWS granule profiles are spherical with smooth surfaces, shown in Figure 2. (A), whilst PWS granules are irregular shapes and fractured, resulting in the rough surface (as seen in Figure. 2 (B)). This result was in agreement with the result of Barua et al. (2022), the modification of elephant foot yam starch caused several surface damages during processing while the native starch granules were spheroidal, ellipsoidal, and polygonal with no obvious signs of damage on the surface. The changes in starch granules before and after the pregelatinization process are due to the increasing granule swelling as the increasing temperature and the presence of water entering the starch granule (Ulfa et al., 2021). The drying process affected the microstructure of pregelatinized starch; oven-drying created a starch granule structure similar to compact rocks (Agama-Acevedo et al., 2018). Oven drying is a convection process in which molecules of water in the solid matrix were removed and transferred to the surface, resulting in shrinkage during the evaporation process of water and changes in the shape and structure of the product (Bonazzi and Dumoulin, 2011).

XRD results

The pregelatinization process using various temperatures and time shows different diffraction patterns of NWS and PWS. The XRD patterns of NWS and PWS can be seen in Figure 3. NWS exhibited peaks (2θ) at 15.07° , 17.78° , 17.85° , 23.03° , 30.05° , 38.17° , 72.46° , and at 72.59° , the PWS shows peaks (2θ) at 15.26° , 17.14° , 22.86° , 44.58° , and 72.61° . However, NWS shows sharper diffraction peaks than PWS. This diffraction pattern shows that NWS has higher crystallinity than PWS. It is due to its amylose content which is lower than PWS and may be correlated to its higher amylopectin content (Kankate et al., 2020). Barua et al. (2022) observed that the peak of the modified elephant foot yam using hot air treatment shows broader peaks at 2θ values of 15.18° , 17.13° , 18.03° , and 23.08° . Whilst Reddy et al. (2014) claimed that XRD peaks for elephant foot yam starch at diffraction angles of 2θ appeared at 17.92° and a strong peak at 23.05° , with a weak peak at 14.7° . The oven-dried process encourages water molecules to move slowly from the pregelatinized starch matrix, a phenomenon that is related with the starch chains' re-crystallization and propagation (Agama-Acevedo et al., 2018).

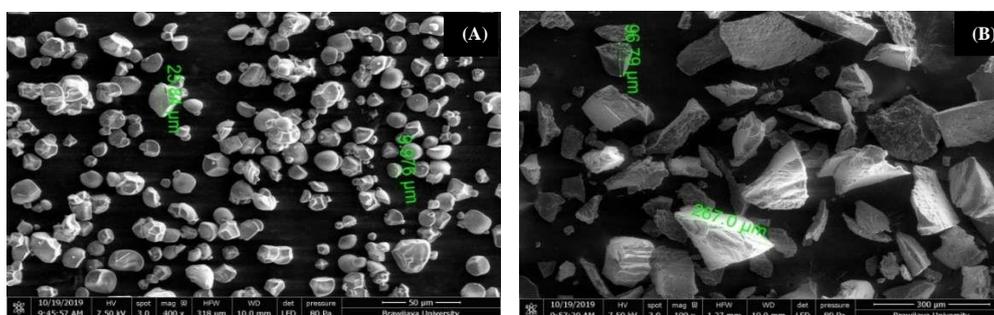


Figure 2. SEM micrographs of native walur starch (A) and pregelatinized walur starch (B); XRD X-Ray Pattern (C) of native walur starch (a) and pregelatinized walur starch (b)

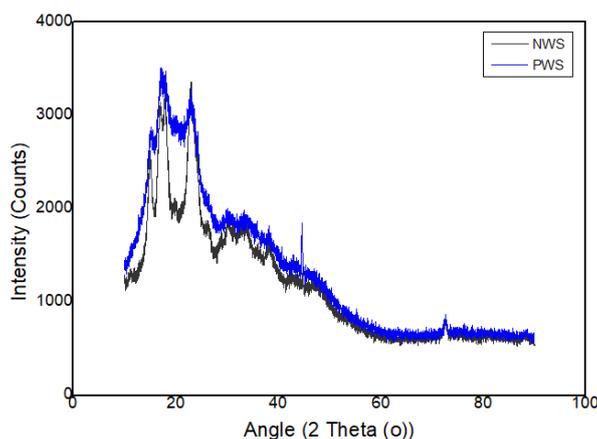


Figure 3. XRD X-Ray Pattern of native walur starch (NWS) and pregelatinized walur starch (PWS)

Conclusion

The swelling, solubility, and WHC of PWS were affected by the temperature and time of the pregelatinization process. The verification experiment supports the predicted design expert for optimizing the pregelatinization processes of PWS, and the results showed that not to be significantly ($P > 0.05$) different. The optimized condition process predicted by the models was at temperature of 87.51°C and 9.71 minutes heating time. Pasting properties of PWS showed higher through, final, and setback viscosity, peak time, and lower breakdown viscosity than the NWS sample. The pasting temperature and peak viscosity of PWS were almost similar to NWS. The NWS had a more crystalline form than PWS on XRD analysis. The SEM analysis revealed that PWS had sharp edge granules, and the smooth surface of NWS granules was ruptured.

Declarations

Conflict of interests The authors declare no competing interests.

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References

- Abbas, K. A., Khalil, S., and Meor Hussin, A. S. (2010) 'Modified starches and their usages in selected food products: A review study', *Journal of Agricultural Science*, 2(2), pp. 90-100
- Abedi, E., Pourmohammadi, K., Jahromi, M., Niakousari, M., and Torri, L. (2019) 'The effect of ultrasonic probe size for effective ultrasound-assisted pregelatinized starch', *Food and Bioprocess Technology*, 12(11), pp. 1852–1862
- Agama-Acevedo, E., Pacheco-Vargas, G., Bello-Perez, L. A., and Alvarez-Ramirez, J. (2018) 'Food hydrocolloids effect of drying method and hydrothermal treatment of pregelatinized hylon VII starch on resistant starch content', *Food Hydrocolloids*, 77, pp. 817–824
- Aktaş, N., and Gerçekaslan, K. E. (2018) 'Effect of pregelatinization and retrogradation on some physicochemical changes of wheat-potato starches', *Agricultural Sciences*, 25, pp. 281–289
- Alam, F., and Hasnain, A. (2009) 'Studies on swelling and solubility of modified starch from taro (*Colocasia esculenta*): Effect of pH and temperature', *Agriculturae Conspectus Scientificus*, 74(1), pp. 45–50
- Alcázar-Alay, S. C., and Meireles, M. A. A. (2015) 'Physicochemical properties, modifications and applications of starches from different botanical sources', *Food Science and Technology*, 35(2), pp. 215–236
- Anggraini, D., Lukman, A., and Teruna, H. Y. (2016) 'Physicochemical properties of phosphate pregelatinized musa balbisiana starch as pharmaceutical excipient', *Pharmacology and Clinical Pharmacy Research*, 1(3), pp. 90–96
- Askari, M., Abbaspour-Gilandeh, Y., Taghinezhad, E., El Shal, A. M., Hegazy, R., and Okasha, M. (2021) 'Applying the response surface methodology (RSM) approach to predict the tractive performance of an agricultural tractor during semi-deep tillage', *Agriculture (Switzerland)*, 11(11), pp. 1-14
- Barua, S., Hanewald, A., Bachle, M., Mezger, M., Prakash, P., and Vilgis, T. A. (2022) 'Insights into the structural, thermal, crystalline and rheological behavior of various hydrothermally modified elephant foot yam (*Amorphophallus paeoniifolius*) starch', *Food Hydrocolloids*, 129, pp. 1-14
- Barua, S., Rakshit, M., and Srivastav, P. P. (2021) 'Optimization and digestogram modeling of hydrothermally modified elephant foot yam (*Amorphophallus paeoniifolius*) starch using hot air oven, autoclave, and microwave treatments', *LWT*, 145, pp. 1-10
- BeMiller, J. N. (2018) *Carbohydrate Chemistry for Food Scientists*. Amsterdam: Elsevier Inc.
- Bonazzi, C., and Dumoulin, E. (2011) 'Quality changes in food materials as influenced by drying processes' in Tsotsas, E., and Mujumdar, A. S. (eds.) *Modern Drying Technology*. German: Wiley - VCH Verlag GmbH & Co. KGaA, pp. 1–20.
- Box, G. E. P., and Wilson, K. B. (1951) 'On the experimental attainment of optimum conditions', *Journal of the Royal Statistical Society. Series B*, 13, pp. 1–45
- Gerçekaslan, K. E. (2020) 'Hydration level significantly impacts the freezable - and unfreezable -water contents of native and modified starches', *Food Science and Technology (Campinas)*, 41(2), pp. 1-6
- Ghodke, S. K., Ananthanarayan, L., and Rodrigues, L. (2009) 'Use of response surface methodology to investigate the effects of milling conditions on damaged starch, dough stickiness and chapatti quality', *Food Chemistry*, 112(4), pp. 1010–1015
- Kankate, D., Panpalia, S. G., Kumar, K. J., and Kennedy, J. F. (2020) 'Studies to predict the effect of pregelatinization on excipient property of maize

- and potato starch blends', *International Journal of Biological Macromolecules*, 164, pp. 1206–1214
- Khare, A. K., Biswas, A. K., Balasubramaniam, S., Chatli, M. K., and Sahoo, J. (2015) 'Optimization of meat level and processing conditions for development of chicken meat noodles using response surface methodology', *Journal of Food Science and Technology*, 52(6), pp. 3719–3729
- Murayama, D., Kasano, M., Santiago, D. M., Yamauchi, H., and Koaze, H. (2014) 'Effect of pre-gelatinization on the physicochemical properties of dry flours produced from 5 cassava varieties of the Philippines', *Food Science and Technology Research*, 20(6), pp. 1131–1140
- Mutaqin, A. Z., Kurniadie, D., Iskandar, J., Nurzaman, M., and Partasasmita, R. (2020) 'Ethnobotany of suweg, *Amorphophallus paeoniifolius*: Utilization and cultivation in West Java, Indonesia', *Biodiversitas*, 21(4), pp. 1635–1644
- Myers, R. H., Montgomery, D. C., and Anderson-cook, C. M. (2016) *Response Surface Methodology: Process and Product Optimization Using Designed Experiments, 4th Edition*. New York: Wiley.
- Neelam, K., Vijay, S., and Lalit, S. (2012) 'Various techniques for the modification of starch and the applications of its derivatives', *International Research Journal of Pharmacy*, 3(5), pp. 25–31
- Niba, L. L., Bokanga, M. M., Jackson, F. L., Schlimme, D. S., and Li, B. W. (2002) 'Physicochemical properties and starch granular characteristics of flour from various manihot esculenta (cassava) genotypes', *Journal of Food Science*, 67(5), pp. 1701–1705
- Odeku, O. A., Schmid, W., and Picker-Freyer, K. M. (2008) 'Material and tablet properties of pregelatinized (thermally modified) Dioscorea starches', *European Journal of Pharmaceutics and Biopharmaceutics*, 70(1), pp. 357–371
- Oladebeye, A. O., Oshodi, A. A., Amoo, I. A., and Karim, A. A. (2013) 'Hydroxypropyl derivatives of legume starches: Functional, rheological and thermal properties', *Starch/Staerke*, 65(10), pp. 762–772
- Purnomo, E. H., Anggraeni, R., Hariyadi, P., Kusnandar, F., and Risfaheri. (2011) 'Reduksi oksalat pada umbi walur (*Amorphophallus campanulatus* var. *Sylvestris*) dan aplikasi pati walur pada cookies dan mie (Oxalate reduction in walur tubers (*Amorphophallus campanulatus* var. *Sylvestris*) and application of walur starch in cookies and noodles)', *Prosiding Perhimpunan Ahli Teknologi Pangan Indonesia (PATPI)*, pp. 5–11 [In Indonesian]
- Reddy, C. K., Haripriya, S., Noor Mohamed, A., and Suriya, M. (2014) 'Preparation and characterization of resistant starch III from elephant foot yam (*Amorphophallus paeoniifolius*) starch', *Food Chemistry*, 155, pp. 38–44
- Roudi, A. M., Salem, S., Abedini, M., Maslahati, A., and Imran, M. (2021) 'Response Surface methodology (RSM)-based prediction and optimization of the fenton process in landfill leachate decolorization', *Processes*, 9(12), pp. 1-20
- Singh, A., and Kumar, K. J. (2020) 'Enhancing delayed release characteristics of chapparada aware seed starch', *International Journal of Biological Macromolecules*, 165, pp. 1431–1437
- Subroto, E., Indiarto, R., Marta, H., and Shalihah, S. (2019) 'Effect of heat - moisture treatment on functional and pasting properties of potato (*Solanum tuberosum* L. var. *Granola*) starch', *Food Research*, 3(2), pp. 469-476
- Suriya, M., Reddy, C. K., and Haripriya, S. (2019) 'Functional and thermal behaviors of heat-moisture treated elephant foot yam starch', *International Journal of Biological Macromolecules*, 137, pp. 783–789
- Suriya, M., Rethina, C., Bashir, M., Koteswara Reddy, C., Harsha, N., and Haripriya, S. (2017) 'Impact of γ -irradiation on physicochemical properties of freeze dried *Amorphophallus paeoniifolius* flour', *Food Chemistry*, 234, pp. 276–284
- Ulfa, G. M., Putri, W. D. R., Fibrianto, K., and Widjanarko, S. B. (2021) 'Optimization studies on pre-gelatinized sweet potato starch influenced by temperature and time', *Food Research*, 5, pp. 25–30
- Varnalis, A. I., Brennan, J. G., MacDougall, D. B., and Gilmour, S. G. (2004) 'Optimisation of high temperature puffing of potato cubes using response surface methodology', *Journal of Food Engineering*, 61(2), pp. 153–163
- Widjanarko, S. B., Amalia, Q., Hermanto, M. B., and Mubarak, A. Z. (2018) 'Evaluation of the effect of yellow konjac flour- κ -carrageenan mixed gels and red koji rice extracts on the properties of restructured meat using response surface methodology', *Journal of Food Science and Technology*, 55(5), pp. 1781–1788
- Witek-Krowiak, A., Chojnacka, K., Podstawczyk, D., Dawiec, A., and Pokomeda, K. (2014) 'Application of response surface methodology and artificial neural network methods in modelling and optimization of biosorption process', *Bioresource Technology*, 160, pp. 150–160
- Yuzammi, A. K., Asih, N. P. S., Erlinawati, I., and Hetterscheid, W. (2017) *The Amorphophallus Indonesia*. Indonesia: Center for Plant Conservation – Botanic Gardens, Indonesian Institute of Sciences (LIPI)
- Zhu, B., Liu, J., and Gao, W. (2017) 'Process optimization of ultrasound-assisted alcoholic-alkaline treatment for granular cold water swelling starches', *Ultrasonics Sonochemistry*, 38, pp. 579–584