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Optimization of temperature and reaction influence on ultrasound-modified sweet potato starch

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Abstract

Native starch has limitations when used directly, such as low solubility, hydration, and stability. Ultrasound as a novel physical technique can overcome these limitations. The response surface method was used to investigate the optimization of temperature and reaction time on ultrasound sweet potato starch. The results show that using 59.58°C for 8.01 mins gives the best products on ultrasound-modified starch. The micrograph of modified starch showed a rougher surface and pores than native starch. These could increase starch hydration and the functional properties of starch. Ultrasound starch is suggested being used as an adsorbent for oil and other bioactive compounds to increase its stability.

1. Introduction

Starch is a polysaccharide and is the most abundant energy source in nature after cellulose (Wang *et al*., 2020). Starch can be used as a thickener, stabilizer, adsorbent, filler, and coating agent, among other things (Butt *et al.*, 2018; Chen *et al.*, 2020). However, due to its low solubility, stability, absorption, and a high tendency to retrograde, starch in its natural form has limitations (Ulfa *et al*., 2020). These weaknesses can be overcome by starch modification.

There are three methods to modify starch, physical, chemical, and enzymatic (Nawaz *et al*., 2020). Chemical modification can take place quickly. However, it leaves chemical residues and raises environmental issues (Lawal, 2019). The enzymatic method is fast and efficient but difficult to control and costly (Zhao *et al*., 2018). Conversely, physical modification is easy and inexpensive, so the use of physical methods tends to increase in popularity (Wang *et al*., 2020). One of the novel and green technology for physical modification is ultrasound.

Ultrasound is an environmentally friendly technology used to modify the functional properties of starch. Ultrasonic mechanical action can occur in liquids by forming cavitation bubbles. Ultrasonic applications benefit from reducing the time and the usage of chemical compounds in reactions (Abedi *et al*., 2019). Ultrasonic can affect swelling, solubility, hydration ability, and starch pasting properties (Jambrak *et al*., 2010; Krishnakumar and Sajeev, 2018; Abedi *et al*., 2019).

Factors such as frequency, intensity, reaction medium, system temperature, duration, botanical nature of starch, and the composition of gases in the atmosphere influence the ultrasound modification (Jambrak *et al*., 2010; Zhu, 2015). Ultrasonics induce cracks and pores in the amorphous part of the starch granule structure, simplifying starch modification by increasing reaction efficiency (Huang *et al*., 2007; Majzoobi *et al*., 2015). Therefore, this study aims to optimize the temperature and reaction time in the ultrasonic process. Sweet potato starch was chosen because of its limited use compared to corn, wheat, cassava, and potato starches. Starch modification could increase the value of sweet potato starch.

2. Materials and methods

2.1 Preparation of modified starch and research design

Sweet potato was obtained from Kulonprogo market, Yogyakarta, Indonesia (7° 38'42"-7° 59'3" South Latitude and 110° 1'37"-110° 16'26" East Longitude). The sweet potatoes were peeled, washed, grated, mixed with water, then precipitated to obtain starch. The starch was oven-dried at 50±2°C for 24 hrs. The starch was stored for further analysis.

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Table 1. The experiment design variables and responses of ultrasound-treated sweet potato starch.

Std	Run	Variables				Responses		
		Code	Actual (°C)	Code	Actual (Mins)	Solubility (%)	Swelling power (g/g)	Water binding capacity (%)
1	8	-1	40	-1	5	6.31	24.75	86.98
2	2	1	60	-1	5	5.44	23.01	78.54
3	11	-1	40	1	15	7.27	25.03	94.28
4	6	1	60	1	15	7.51	36.75	96.74
5	3	-1.41	35.86	0	10	5.52	28.75	84.51
6	5	1.41	64.14	0	10	4.44	33.35	86.36
7	7	0	50	-1.41	2.93	3.72	22.71	82.44
8	13	0	50	1.41	17.07	11.52	30.49	96.79
9	1	0	50	0	10	10.76	38.59	97.72
10	12	0	50	0	10	10.71	38.98	105.06
11	9	0	50	0	10	13.36	39.58	104.34
12	4	0	50	0	10	10.13	36.69	102.36
13	10	0	50	0	10	14.55	41.55	97.84

The ultrasound process was carried out based on Jambrak *et al*. (2010) method with slight modifications. One hundred grams of starch was added with 100 mL of distilled water and homogenized. The starch suspension was given ultrasonication with an ultrasound bath (JP-010s, Skymen, China) with the temperature and time according to the experimental design. After ultrasonication, the starch suspension was oven-dried at 50±2°C and stored in dark bottles with silica gel for analysis.

The response surface method with a central composite design was used as an experimental design. The factors evaluated were ultrasonic temperature (50-60°C) and time (5-15 mins) using a Design Expert (11.0, Stat-Ease, USA). Each treatment was repeated at five center points, four times factorial, and four times axial for thirteen experiments (Table 1). Verification results used targeted to as the criteria designed according to commercial potato starch.

2.2 Physicochemical analysis

Kaur *et al*. (2011) methods were used to analyze starches' solubility and swelling power. A starch slurry (1%) was made in a centrifuge tube and heated in a water bath maintained at 90°C for 30 mins. After that, the suspension was centrifuged at 3200 rpm for 10 mins, and the liquid fraction was separated. The liquid fraction was drained and weighed and calculated using equation (1). The sediment part was weighed and calculated with equation (2).

$$\text{Solubility (\%)} = \frac{\text{weight of soluble starch (g)}}{\text{weight of starches sample (db)(g)}} \times 100\% \quad (1)$$

$$\text{Swelling Power (g/g)} = \frac{\text{weight of sediment paste (g)}}{\text{weight of starches sample (db)(g)}} \quad (2)$$

The water binding capacity of starches was measured by Kaur *et al*. (2011) method with slight modification on the stirring time. The suspension of starch (2.5 g) and 20 mL distilled water in the tube was stirred for 1 hr and centrifuged at 3000 rpm for 10 mins. Excess water inside the tube was removed, and the precipitated part was weighed to calculate the equation (3).

$$\text{Water Binding Capacity (\%)} = \frac{\text{weight of residual starch (g)}}{\text{weight of sample (g)}} \times 100\% \quad (3)$$

The total starch content was measured by the acid hydrolysis method by Pirt and Whelan (1951), amylose content measured following the method by Juliano (1981), and color analysis using the CIE whiteness index by Ulfa *et al*. (2021).

2.3 Morphological analysis

The micrography of starches was examined using Scanning Electron Micrography (TM3000, Hitachi, Japan). The samples were coated with palladium in a vacuum environment.

2.4 Pasting properties analysis

A Rapid visco analyzer machine (RVA-TecMaster, Newport Scientific, Australia) was used to analyze the pasting characteristics of starches.

2.5 Atomic bonding analysis

The atomic bonding was analyzed using Fourier Transform Infrared (8400S, Shimadzu, Japan). The regions scanned by FTIR were between 400-4000 cm^{-1}.

3. Results and discussion

The solubility, swelling power, water binding capacity, amylose, and total starch content of native and treated starch are reported in Table 2. Due to the temperature and time, the solubility, swelling power, and water binding capacity parameters increased in treated starch samples (Figure 1). The software suggested the quadratic model as the model of experiments resulting in the responses equations (Equations (4), (5), and (6)). The optimal ultrasonication conditions are 59.58°C for 8.01 mins.

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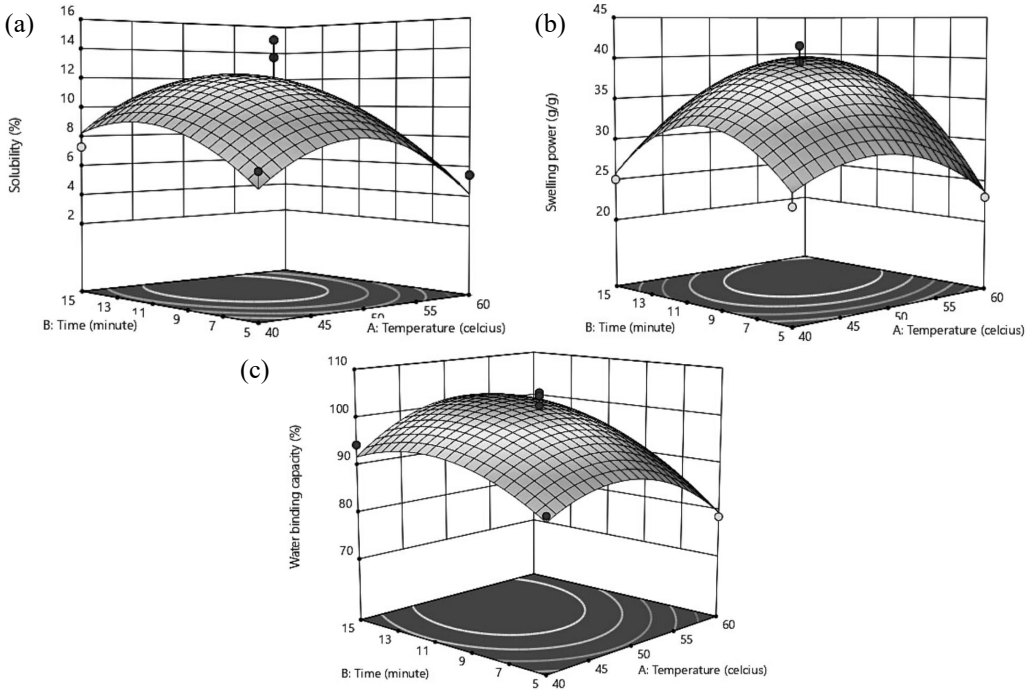

Figure 1. The 3D graphs of (a) solubility; (b) swelling power; (c) water binding capacity of ultrasound-treated starch. The quadratic models suggested by the software are presented in these graphs.

$$\text{Solubility (\%)} = +9.79 + 1.00 \times A - 0.19\ B - 0.35 \times A \times B - 3.07 \times A^2 - 1.56 \times B^2 \quad (4)$$

$$\text{Swelling power (g/g)} = + 39.08 + 2.08 \times \text{A} + 3.1 \times \text{B} + 3.36 \times A \times \text{B} + 4.40 \times A^2 - 7.72 \times B^2 \quad (5)$$

$$\text{Water binding capacity (\%)} = + 111.84 + 3.56 \times \text{A} + 1.23 \times B - 4.27 \times A \times B - 12 \times A^2 - 7.72 \times B^2 \quad (6)$$

Where A is the temperature (°C), B is the time (mins). It was reported that the ultrasound increased the solubility, swelling power, and water binding capacity (Jambrak *et al*., 2010; Manchun *et al*., 2012; Krishnakumar and Sajeev, 2018; Wang *et al*., 2020). The increase of solubility and swelling ability is obtained with the temperature rise, probably due to the reduction of starch crystallinity. These findings are also in accordance with Ulfa *et al*. (2020). The reduction of crystallinity will facilitate the water ingress, binding the water into a free hydroxyl group with hydrogen bonds and increasing starch hydration ability (Abedi *et al.*, 2019; Ulfa *et al*., 2021).

Table 2. The physicochemical analysis of native and ultrasound-treated starches.

Parameters	Native starch	Ultrasound-treated starch
Solubility (%)	1.54±0.01	5.93±0.24
Swelling power (g/g)	16.26±0.52	32.17±0.99
Water binding capacity (%)	76.07±0.66	90.01±1.67
Amylose (%)	42.64±0.13	41.90±0.42
Total starch (%)	93.92±1.60	92.94±0.03
Whiteness index	85.79±0.03	85.39±0.04

A reduction in amylose and total starch content of treated starch increases solubility. These findings are in accordance with the results reported by Abedi *et al*. (2019). Several studies found that the solubility of starch have an inversely proportional relationship with amylose content (Hasmadi *et al*., 2021). Other reports have shown the significant role of amylose in the starch's ability to swell and absorb water (Krishnakumar and Sajeev, 2018; Abedi *et al*., 2019).

Ultrasonic starch had a lower whiteness index when compared with native starch. These findings are also shown in several studies about the sonication of tuber starch (Krishnakumar and Sajeev, 2018) and sweet potato and wheat flour (Cui and Zhu, 2020). This reduction is probably due to the development of light brown pigments by Maillard and caramelization reactions (Abedi *et al*., 2019). In addition, the possibility of some impurities bonding with starch molecules can happen during sonication due to starch disintegration. These phenomena will result in the reduction of the whiteness index (Krishnakumar and Sajeev, 2018).

Ultrasonic treatments can affect the starch at various temperatures and times through three particular mechanisms: (1) damage the starch granule with the formation of pit and channels; (2) change the granular rearrangement by interrupting the C-C bonds of starch; and (3) solubilize the aggregates and swollen granule as well as the ghost granules (Zhu, 2015). The higher the temperature and the longer the heating time will have more degradation impact (Krishnakumar and Sajeev, 2018).

Micrographs of native and treated starches are presented in Figure 2. Ultrasound starch showed no significant change in granule size with a starch diameter of 7-23 μm compared to native starch of 4-24 μm. Several studies have shown that ultrasonication does not

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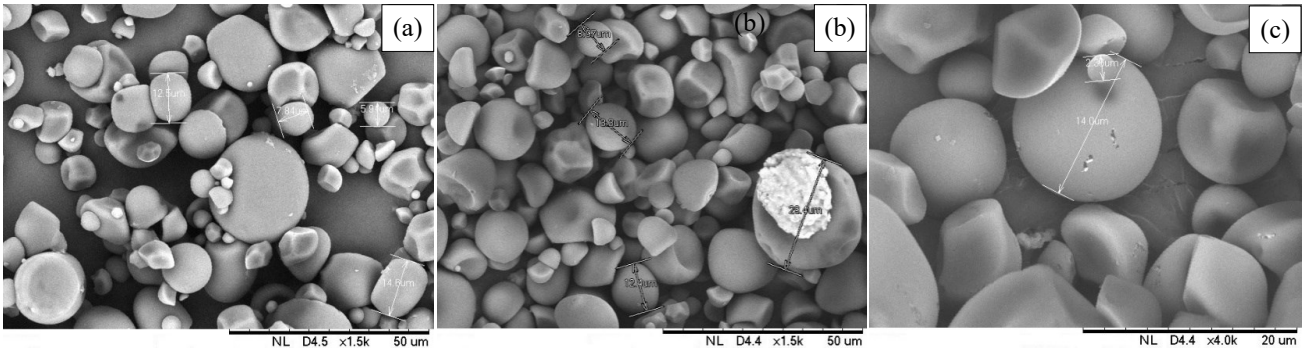

Figure 2. The micrographs of (a) native (1500×), (b) ultrasound-treated (1500×), (c) ultrasound-treated (4000×) starches.

change granule size (Huang *et al.*, 2007; Sujka and Jamroz, 2013; Zheng *et al.*, 2013).

However, a rougher and more porous surface appears on ultrasonic starch (Figure 2 (c)). These surface changes are probably due to the ultrasonic treatment that damages starch granules mechanically. The damage occurs through the collapse of the cavitation bubbles. This change will induce high surface pressure and surface fluid velocities. The shear forces of pressure and fluid movement can break the starch polymer chains and disrupt the granules (Zheng *et al*., 2013).

Pores that appear on the surface of the starch granules appear and increase the starch surface area. This can increase the absorption capacity of starch so that the reagent is more comfortable entering into the starch granule and accelerating chemical reactions. Therefore, ultrasonication can increase efficiency (Bemiller and Huber, 2015; Zhang *et al*., 2019), and the usage of ultrasound starch as an adsorbent is recommended.

There is no change in the atomic bonding of starch characterized by FTIR (Figure 3). There were no changes or additional peaks after sonication. These findings are also shown in the dual-sonication of sweet potato starch (Zheng *et al*., 2013). The ultrasonic treatment does not cause changes in the starch molecular structure. Hence, the ultrasound-treated starch functional groups are similar to native starches. However, a change in peak absorption intensity decreased after ultrasonic treatment. This change is probably due to the sonication and will increase the absorption of infrared and lowering the percent transmission (Lawal *et al*., 2015; Ulfa *et al.*, 2020).

The pasting characteristics of native and ultrasound-treated starches are presented in Figure 4. The final and setback viscosity of ultrasound starch was higher than the native starch. At the same time, the breakdown viscosity was lower than native starch. These findings also happened in the ultrasonication of corn (Zhang *et al*., 2005) and cassava (Krishnakumar and Sajeev, 2018) starches. The rise in the pasting temperature of ultrasound starch indicates that granule swelling happened at higher temperatures before the disruption. The higher final and setback viscosity value indicated that starch could form a rigid gel and be more resistant to shear forces. This functionality was significantly influenced by sonication temperature and time (Krishnakumar and Sajeev, 2018; Ulfa *et al*., 2021).

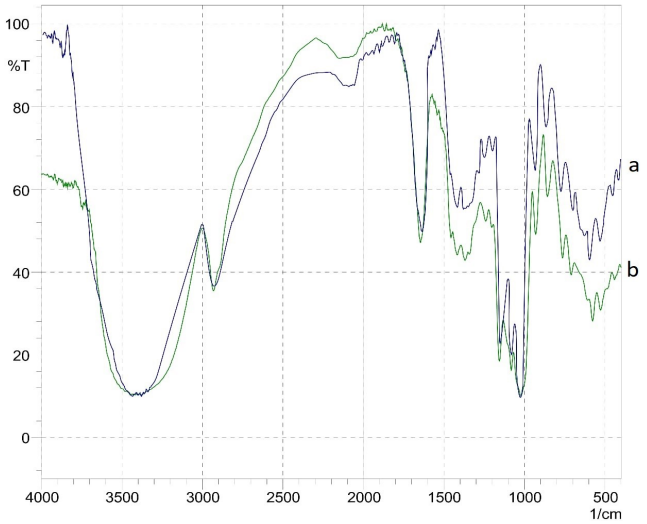

Figure 3. The infrared graph of (a) native and (b) ultrasound-treated starches using FTIR.

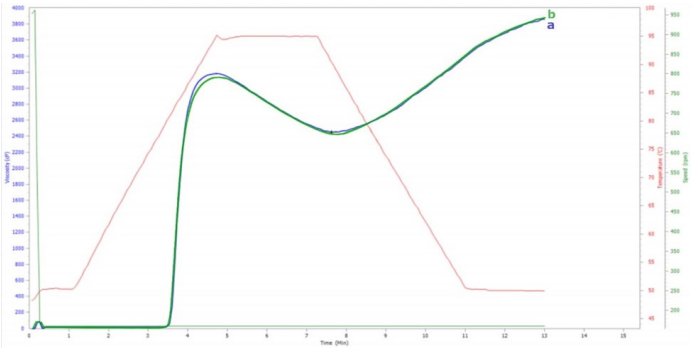

Figure 4. The pasting characteristics of (a) native and (b) ultrasound-treated starches using RVA.

4. Conclusion

The structure and physicochemical properties of ultrasound starch using 59.58°C for 8.01 min yield the best results on modified starch. The modified starch micrograph revealed a rougher surface and pores than native starch, indicating a higher surface area. This suggests the usage of it as an adsorbent for sensitive elements and other bioactive compounds to increase its stability. However, the effect of ultrasonication on starch

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has not been studied enough. Other parameters such as ultrasound frequency and power should be optimized to reduce the adverse effect of free radicals formed during sonication.

Conflict of interest

The authors declare there is no conflict of interest.

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