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Optimization of Breadfruit (*Artocarpus altilis*) via Succinylation Reaction on the Effect of Time Duration and Temperature

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ABSTRACT

Native breadfruit starch exhibits limitations such as poor water solubility, low thermal stability, and restricted functional properties, which hinder its direct application in industrial fields. In this study, modified starch was synthesized using a succinylation reaction between breadfruit (*Artocarpus altilis*) starch and succinic acid on a pyridine catalyst with time variations of 3, 3.5, and 4 h, and temperature variations of 95°C, 105°C, and 115°C. From the isolation of breadfruit starch, a yield of 7.5% was obtained. The modified succinic starch was analyzed for functional groups using an FT-IR spectrophotometer, SEM, and TGA, and the degrees of substitution, solubility, and swelling power were then determined. The formation of succinylated starch was confirmed by FT-IR spectra, which exhibited a characteristic C=O ester vibrational band at 1,692 cm^{-1} and a supporting peak at 1,200 cm^{-1} corresponding to the C-O-C ester functional group. The DS values were measured across all variations of time and temperature, with the highest DS value of 0.5293 observed at 3.5 h and 105°C. Both swelling power and solubility increased with longer reaction times and higher temperatures. Thermal analysis indicated that the starch samples experienced significant degradation at 400.6°C, with a mass loss of 82.9%. SEM images revealed that the succinylation reaction caused fragmentation of starch granules, indicating a structural modification of the starch. Overall, the transformation of native breadfruit starch into succinylated starch enhanced its functional properties, demonstrating its potential as an environmentally friendly material for bioplastic applications. Further investigations are recommended to assess the mechanical performance and biodegradation behavior of the resulting bioplastics in order to comprehensively evaluate their potential for commercial application.

HIGHLIGHTS

- Modification of breadfruit starch by succinylation method with pyridine catalyst
- The modified succinic starch was analyzed for functional groups using FT-IR spectrophotometer, SEM, TGA, followed by determination of degree of substitution, solubility, and swelling power.
- Greater water absorption causes swelling power to increase

1. INTRODUCTION

Starch, a semi-crystalline biopolymer, is a highly versatile raw material with a wide range of applications, including as a staple food in human diets, food additives, biodegradable packaging materials, and more. For starch applications, the structure (semi-crystalline lamellae, crystalline structure, and molecular structure) and properties (swelling index, thermal properties, adhesive properties, and digestibility) of various starches have been extensively studied (Huang et al., 2016; Tan et al., 2015; Wongsagonsup et al., 2008). Specifically, corn starch, rice starch (Deng et al., 2014), cassava starch (Mei et al., 2015), and potato starch have been well-researched. However, the increasing demand for starch from modern industries has generated intense interest in new and underutilized polysaccharide sources, opening opportunities for other plants, given

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the high starch demand. There are also less common starches, such as breadfruit starch, that are worth investigating for the development of new starch-based products with enhanced properties (Tan et al., 2017).

Breadfruit (*Artocarpus altilis*) is a tropical fruit native to Indonesia, the South Pacific, and the Caribbean, and belongs to the Moraceae family (Wang et al., 2011). Breadfruit is a popular starch-producing fruit widely developed in Indonesia. Its high carbohydrate content makes it a valuable source for starch production. Starch extracted from breadfruit yields 18.5 g/100 g with a purity of 98.86% and consists of 27.68% amylose and 72.32% amylopectin (Fatimah Zuhra et al., 2022). Due to the fruit's poor fresh storage quality, converting it into flour and starch offers a more stable form and enhances its versatility. Since ancient times, native breadfruit starch has been used as a raw material for preparing various products (Alam et al., 2024). To expand its applications, several physical and chemical modifications of breadfruit starch, such as acetylation, oxidation, HMT (heat moisture treatment), and fermentation, have been studied recently (Adebowale et al., 2005). However, the digestibility of breadfruit starch is rarely reported. Haydersah et al. (2012) studied the digestibility of breadfruit starch and found that fermentation can increase its resistant starch content.

Native starch is not soluble in water and contains granules whose size, composition, physicochemical, and functional properties depend on plant characteristics and environmental conditions. Native starch from various plant sources generally has properties that limit its use in different food products. Improvements in the physical and chemical properties of native starch can be made, among other ways, through starch modification (Volkert et al., 2010). Modified starch is starch whose hydroxyl groups have been altered through a reaction or by changing its original structure. Starch is treated with specific processes to produce better properties, improving its previous characteristics to meet industrial needs. Modified starch has properties that native starch does not, these include higher brightness (whiter starch), lower retrogradation, lower viscosity, more apparent gel formation, softer gel texture, lower tensile strength, starch granules that break more easily, higher gelatinization time and temperature, and lower time and temperature for starch granules to break.

Starch modification can be carried out chemically through cross-linking, oxidation, etherification, esterification, or substitution, as well as through a combination of these methods (dual and more modification). Modification via esterification is one of the best starch modifications used. Modification through esterification can slow the rate of starch retrogradation, which is caused by the inhibition of hydrogen bond formation between amylose and amylopectin molecules by the ester groups formed. The advantages of starch modified through esterification include reduced gelatinization temperature, increased viscosity, higher water-binding capacity, and a clearer paste. Compared to cross-linked starch, esterified starch still experiences a decrease in viscosity during the heating process (unstable under heating) and is less resistant to acidic conditions (Zhang et al., 2009; Hamid et al., 2024d). The esterification method using Succinic Acid catalyst has been widely discussed to become succinylated starch. The modification of starch through succinylation has become a significant area of research over the past decade. Succinylated starch is widely recognized as an essential stabilizer due to its surface-active properties. This type of starch displays distinctive features, as its hydrophilic regions acquire hydrophobic traits by introducing octenyl groups, giving the molecule amphiphilic properties. The stabilization mechanism of succinylated starch leverages both the hydrophobic interactions and steric hindrance provided by the succinyl group. Succinylated starch can be further utilized in frozen canned food products and flavor encapsulation materials (Błaszczak et al., 2007; Hamid et al., 2024c).

Then the research conducted by Sri Haryani Anwar is the synthesis of breadfruit pat with succinic acid getting FTIR results that, the peak at 1,720 cm^{-1} is the C=O stretch vibration of the ester carbonyl group, while the peak at 1,560 cm^{-1} is related to the $RCOO^-$ carboxylate stretch vibration (Anwar et al., 2020a). These results indicate that the hydroxyl group in starch is substituted with the carbonyl and carboxyl ester groups of succinic acid. Fourier transform infrared spectroscopy (FTIR) showed that the starch modification was successful (Degree of Substitution, 0.0241) (van der Burgt et al., 2000). The novelty of this research lies in optimizing the preparation of breadfruit (*Artocarpus altilis*) based materials through succinylation chemical reaction utilizing succinic acid as a catalyst, which has not been widely explored in the context of natural biomaterial modification (Rumahorbo et al., 2023). This study specifically highlights the effect of reaction time and temperature on the efficiency of the succinylation process, which aims to improve the physicochemical and functional properties of breadfruit starch. With this approach, it is expected to obtain breadfruit starch derivative products that have better thermal stability, increased solubility, and wider applicative potential in the fields of pharmaceuticals, food, and bioplastics. This innovation provides a new contribution to the development of local biomaterials that are environmentally friendly and have high added value through controlled chemical modification techniques (Chabib et al., 2025; Fatimah Zuhra and Ginting, 2013).

Based on the background described above, the researcher was interested in modifying starch through a succinylation reaction between breadfruit starch (*Artocarpus altilis*) and succinic acid using pyridine as a catalyst, with variations in time and temperature. The succinylated starch was tested by analyzing functional group changes using FT-IR spectroscopy, morphological analysis using SEM, temperature change analysis using TGA, and determining the Degree of Substitution, swelling power, and solubility. This modification aims to produce starch that meets industrial needs.

2. METHODOLOGY

2.1 Materials

Breadfruit was taken from Sumatera Utara Province, Indonesia, and serves as a doping source for starch (Hamid et al., 2024b). Succinic Acid, Pyridine, Ethanol, Hydrochloric Acid, Sodium Hydroxide, and distilled water were bought from Merck in Germany.

2.2 Methods

2.2.1 Isolation of breadfruit starch

First, the breadfruit starch is peeled and the fruit stalk is removed in isolating breadfruit starch is to peel and remove the fruit stalk, then wash it until it is free of dirt and sap. The breadfruit was cut into pieces, pureed using a blender, and squeezed through a gauze filter to get the starch. This was left for 24 h until the starch settled, and then the liquid was washed several times with water until it was clear. The starch obtained was dried in an oven at 45°C for 24 h. The sample was then mashed, sieved, and weighed. Subsequently, it was analyzed using Fourier Transform Infrared (FT-IR) spectroscopy and Scanning Electron Microscopy (SEM) (Hamid et al., 2024a).

2.2.2 Modification of breadfruit starch with succinic acid

The following steps were used in the preparation of breadfruit starch modification using succinylation reaction. First, 10 g of breadfruit starch was suspended in 50 mL of pyridine and put into a 250 mL threeneck flask. Then, it was stirred at 85°C for two h, and 4 g of succinic acid was added and refluxed while stirring for 3.5 h at 105°C. Then, it is cooled at room temperature, washed with distilled water three or more times until a normal pH (7) is obtained, and washed with 70% alcohol twice to ensure that impurities were removed. Then, the starch was dried at 40°C for 24 h and filtered with a 100 mesh sieve. The same procedure was carried out for 3 h and 4 h time variations. Then, it was analyzed by FT-IR spectroscopy, and the degree of substitution was calculated. Then, the highest value of the degree of substitution is carried out the same procedure with temperature variations of 95°C, 105°C, and 115°C. Furthermore, it was characterized by FT-IR spectroscopy, SEM, solubility, and swelling power.

2.2.3 Degree of substitution (DS)

The degree of substitution was determined from the modified breadfruit starch samples using the modified titration method. First, 0.5 g of starch succinate was weighed and placed into an Erlenmeyer flask. Then, 30 mL of distilled water and 15 mL of 0.5 M NaOH were added. The mixture was heated at 30°C until fully dissolved. After that, two drops of phenolphthalein (PP) indicator were added, and the solution was titrated with 0.1 M HCl until it turned colorless (Zuhra et al., 2025). The degree of substitution was then calculated using Equation (1).

$$\text{Degree of substitution (DS)} = \frac{162A}{4300-42A} \quad (1)$$

Where; A=starch content (%).

2.2.4 Fourier transform infrared (FTIR) spectrophotometer

Fourier-transform infrared spectroscopy (FTIR) analysis was performed using a Shimadzu spectrometer. Spectral visualization and processing were carried out using Spectragryph software for optical spectroscopy, version 1.2.13, developed by Dr. Friedrich Menges (Copyright 2019-2020). All spectra were recorded in the range of 4,000-400 cm^{-1}, with an average of 32 scans and a resolution of 8 cm^{-1} (Nasr et al., 2020).

2.2.5 Scanning electron microscopy (SEM)

The surface morphologies of the native and modified starch granules were examined using a scanning electron microscope (SEM) model Phenom Pro Desktop SEM and JEOL JSM-IT200. All samples were carefully mounted on aluminum stubs using double-sided conductive carbon tape and coated with a thin layer of gold to enhance electrical conductivity. The observations were conducted under a high-vacuum mode at an accelerating voltage of 25 kV. Morphological features of the starch granules were captured at a magnification of 5.000 times to allow for detailed visualization of surface texture, granule integrity, and any structural alterations resulting from the modification process (Humaidi et al., 2024).

2.2.6 Thermogravimetric analysis (TGA)

Starch sample thermal decomposition was determined using a Pyris-1 DSC apparatus (PerkinElmer DSC 4000, Shelton, USA). Samples were heated between 30 and 650°C at 10°C/min in an inert atmosphere of nitrogen flowing at 20 mL/min (Oderinde et al., 2020).

2.2.7 Determination of swelling power and solubility

Swelling power and solubility of succinate starch can be used in the method used to cut (Zuhra et al., 2020). Starch is suspended with distilled water (1%, w/v) in a test tube of known weight. Then, it was heated in a water bath at a temperature of 95°C for 30 min and then cooled to room temperature (±27°C). The starch suspension was centrifuged at 5.000 rpm for 15 min to separate the residue and supernatant. The supernatant (10 mL) is dried to constant weight at a temperature of 110°C. The residue from obtained from drying the supernatant shows the amount of starch dissolved in water (%). The residue and water retained after centrifugation ware then weighed. The swelling ability of starch (based on dry weight) was determined as follows is Equations (2) and (3):

$$\text{Swelling of starch} = \frac{\text{Weight of paste}}{\text{Weight of dry sample}} \tag{2}$$

$$\text{Solubility} = \frac{\text{W2} - \text{W1}}{\text{Weight of starch}} \times 100\% \tag{3}$$

Where; W_1=mass of dry test tube + dry starch; W_2=mass of dry test tube + wet starch.

3. RESULTS AND DISCUSSION

3.1 Mechanism

Succinic starch was obtained from the succinylation reaction between breadfruit starch and succinic acid. Succinylation of breadfruit starch was carried out in stages. While at the stage of dissolving breadfruit starch with pyridine. This stage aims to smooth the starch granules so that they can react with succinic acid. Pyridine also serves as a catalyst that can activate the hydroxyl group. After reaching a temperature of 85ºC, succinic acid is added, forming a yellowish gel-shaped starch succinate mixture. The reaction pathway for the succinylation of breadfruit starch with succinic acid is depicted in Figure 1.

The result of the succinylation reaction will product the main product, starch succinate. The gel-shaped succinic starch mixture was then added with 70% alcohol and distilled water and dried in an oven. The resulting succinic starch has physical properties of yellowish color, is lump-shaped, and is more hydrophilic than natural starch. In the reaction mechanism of starch succinate formation, secondary C atoms are substituted by succinic acid because secondary OH groups, especially C-2 OH groups, are more reactive than primary OH groups. The reactivity of OH group C-2 is 60-65%, and OH group C-6 is 15-20% (Burgt et al., 2000).

3.2 Degree of substitution analysis

In this study, the DS results obtained at various times include a 3 h period with a value of 0.5159, then a 3.5 h period with a value of 0.5293, and finally, a 4 h period with a value of 0.4949. Based on the volume of HCl used in the titration, it can be seen that the value of the degree of substitution increases in the 3-h and 3.5-h time variations, namely from 0.5159 to 0.5293, but there is a decrease in the 4-h time variation with a value of 0.4949. Increase in heating time increased he solubility and the DS value of the starch. The decrease in DS value in the 4 h time variation was due to the hydrolysis reaction that decreased the reaction efficiency (Bhosale and Singhal, 2006). In the succinylation process of breadfruit starch, it is known that the highest reaction efficiency is obtained with a reaction time of 3.5 h.

In the temperature variation study, the degree of substitution (DS) obtained at 95°C was 0.4606, increased to 0.5293 at 105°C, and slightly decreased to 0.5201 at 115°C. As shown in Figure 2, the volume of HCl used during titration indicates a trend consistent with the DS values: the DS increased with temperature from 95°C to 105°C, but a slight decline was observed at 115°C. The initial increase in DS with rising temperature is attributed to the enhancement in reaction kinetics; higher temperatures increase molecular motion and collision frequency, thereby accelerating the succinylation reaction (Chi et al., 2008). However, at excessively high temperatures, such as 115°C, partial degradation or structural disruption of the starch granules may occur, which can negatively affect the efficiency of the substitution reaction. Based on these findings, the optimal succinylation condition was achieved at 105°C, where the highest DS and reaction efficiency were observed. When combined with the optimal reaction time of 3.5 h, this temperature condition yielded the most effective modification of breadfruit starch. High temperatures cause starch to undergo gelatinization, leading to the formation of aggregates that hinder the succinylation reaction and reduce its effectiveness (Dewi et al., 2022).

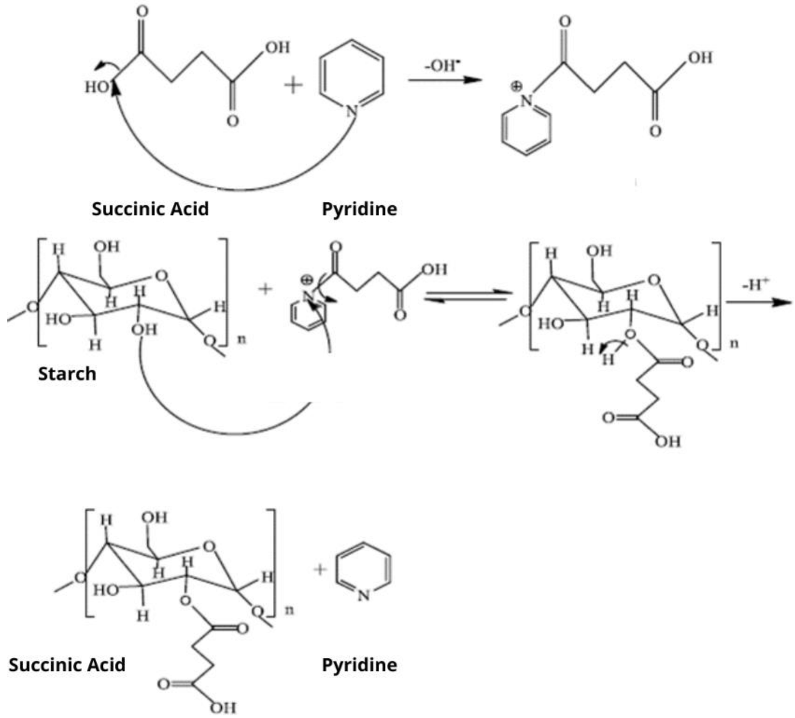

Figure 1. Reaction mechanism for the formation of modified starch

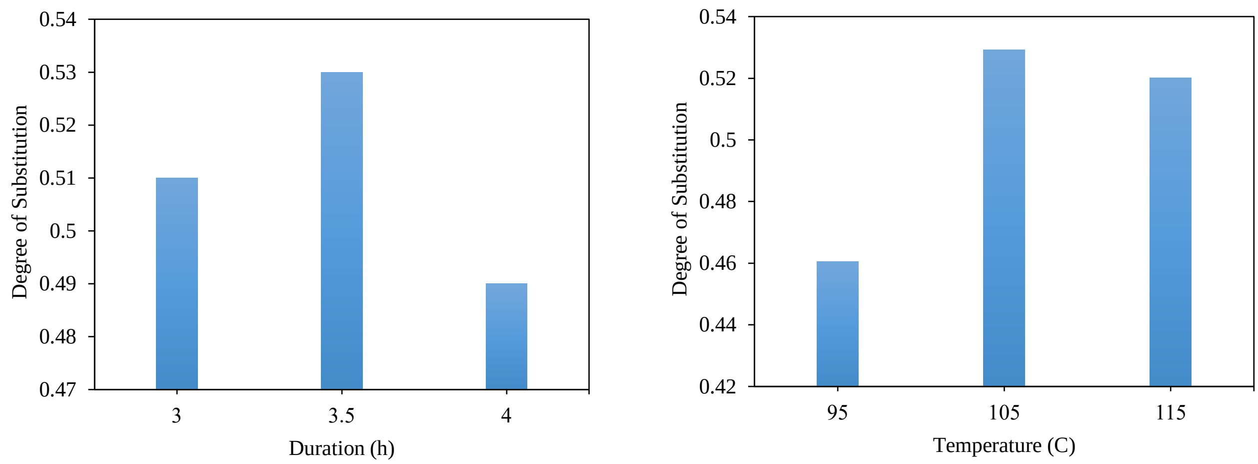

Figure 2. Degree of substitution value of modified breadfruit starch in different durations and the temperature of the reaction.

3.3 FTIR Analysis

FT-IR spectrophotometer test on breadfruit starch can be seen in Figure 3, which shows a spectrum with a peak vibration in the 3,265 cm^{-1} wave number region, indicating the -OH group. At wave number 2,922 cm^{-1}, the absorption shows the C-H alkane (-CH_3) stretching vibration. The absorption at wave number 1,640 cm^{-1} comes from rocking vibrations of -OH bound to water molecules contained in starch through hydrogen bonds the absorption band in the 1,334-1,500 cm^{-1} range shows bending vibrations on C-H. According to Chen et al. (2019), the area between 1,340 cm^{-1} to 1,500 cm^{-1} is the absorption area of rocking vibrations of C-H. The absorption band at wave number 1,148 cm^{-1} shows C-O stretching vibrations. Wave number 1,148 cm^{-1} shows the absorption of C-O stretching vibrations bound to the secondary alcohol hydroxyl group.

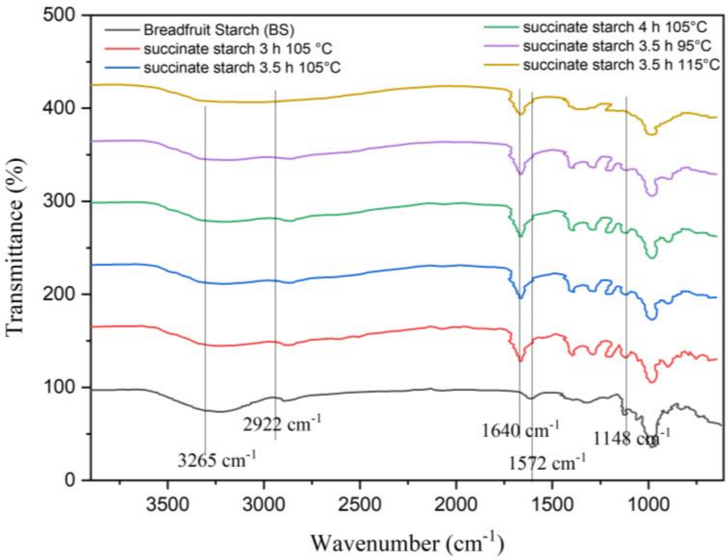

Figure 3. FT-IR spectra of isolated breadfruit starch (a), succinate starch 3 h 105°C (b), succinate starch 3.5 h 105°C (c), succinate starch 4 h 105°C (d), succinate starch 95°C 3.5 h (e), succinate starch 115°C 3.5 h (f).

The comparison of spectra between breadfruit starch (Figure 3) with succinic starch with time variation (Figure 3) showed a change in wave absorption bands, namely in the FT-IR spectrum of succinic starch with time variation showing the C=O ester group at 3 h wave number 1,692.2 cm^{-1} with low intensity, 3.5 h wave number 1,692.2 cm^{-1} with high intensity, 4 h wave number 1,692.2 cm^{-1} with low intensity. The comparison of the spectrum between breadfruit starch and succinic starch with temperature variation, shows a change in the absorption band, namely in the FT-IR spectrum of succinic starch with temperature variation showing the C=O ester group at 95°C wave number 1,692.2 cm^{-1} with medium intensity, at 105°C wave number 1,692.2 cm^{-1} with high intensity, at 115°C wave number 1,692.2 cm^{-1} with medium intensity. The band occurring at 1,572 cm^{-1} corresponds to the asymmetric stretching of the carboxylate RCOO group vibrations that are characteristic of esterified starch. In addition, the intensity of the absorption peaks around 1,692 cm^{-1} and 1,572 cm^{-1} increases as the degree of substitution increases, indicating that more OS groups are inserted into the starch (Hao et al., 2019). The higher intensity of the FT-IR spectrum indicates the increasing effectiveness of succinylation and the higher degree of substitution. In this study, the degree of substitution increased and decreased according to the intensity of the FT-IR spectrum.

3.4 Swelling power and solubility analysis

This study examined the swelling power and solubility of breadfruit starch and succinylated starch, as shown in Table 1. At the treatment condition of 3.5 h at 115°C, the starch sample exhibited a swelling power of 9.34% and a solubility of 57.32%. The increase in starch solubility is attributed to the higher heating of the starch suspension, which leads to the depolymerization of starch molecules, particularly amylose (Ojogbo et al., 2020). Depolymerized amylose consists of shorter and more linear chains that are more readily soluble in water. As a linear polymer, amylose naturally tends to dissolve in water, and this tendency increases when the molecular structure is simplified due to thermal degradation. Therefore, the higher the heating temperature, the greater the extent of amylose breakdown, resulting in increased solubility of the starch. On the other hand, the increase in swelling power is related to a lower amylose content or a higher proportion of amylopectin in the starch. Amylopectin is predominantly located in the amorphous regions of starch granules. These amorphous regions are less dense and more porous, allowing water to penetrate more easily and causing the granules to swell. Thus, higher heating temperatures enhance the swelling power of starch by facilitating water absorption through the amorphous regions rich in amylopectin (Chen et al., 2020).

Table 1. Results of swelling power and solubility determination

Sample	Swelling power (%)	Solubility (%)	Sample	Swelling power (%)	Solubility (%)
Breadfruit starch	8.7646	29.80	-	-	-
3 h 105°C	8.8730	42.08	3.5 h 95°C	8.8372	30.88
3.5 h 105°C	9.0952	49.02	3.5 h 105°C	9.0952	49.02
4 h 105°C	10.5817	59.02	3.5 h 115°C	9.3449	57.32

Then, both swelling power and solubility of starch are strongly influenced by the molecular structure of starch, particularly the interactions between amylose and amylopectin, as well as the structural changes induced by thermal treatment. Succinylation modification alters these intermolecular hydrogen bonds by introducing bulky succinyl groups, which disrupt the native compact structure of starch. As a result, the molecular arrangement becomes more open and hydrophilic, thereby enhancing the starch's ability to absorb water and dissolve more easily. This structural disruption contributes significantly to the observed increases in both swelling power and solubility. The amorphous part is the part that absorbs water more efficiently. The more amylopectin in starch, the wider the amorphous area will be, so water absorption will be more excellent. It is known that swelling power in starch is influenced by water absorption. The greater the water absorption, the increased swelling power (Amari et al., 2021).

3.5 SEM analysis

SEM analysis was conducted to determine the modified starch compounds' morphology. In this study, SEM tests were carried out for breadfruit starch and succinate starch, which had the highest DS value, namely succinate starch with a time variation of 3.5 h at 105°C. Succinate reactions can change the structure of starch granules. This can be seen in succinic starch with a broken granule structure because breadfruit starch reacts with succinic acid compared to natural breadfruit starch granules. The granule shape of breadfruit starch and succinic starch is different at 5.000 times magnification, where the breadfruit starch granules are more rounded, while the succinic starch granules are more broken. This shows that adding succinic acid affects the shape of the starch granules (Luo et al., 2024). The morphology of breadfruit starch is shown in Figure 4(a), and the morphology of succinic starch is shown in Figure 4(b).

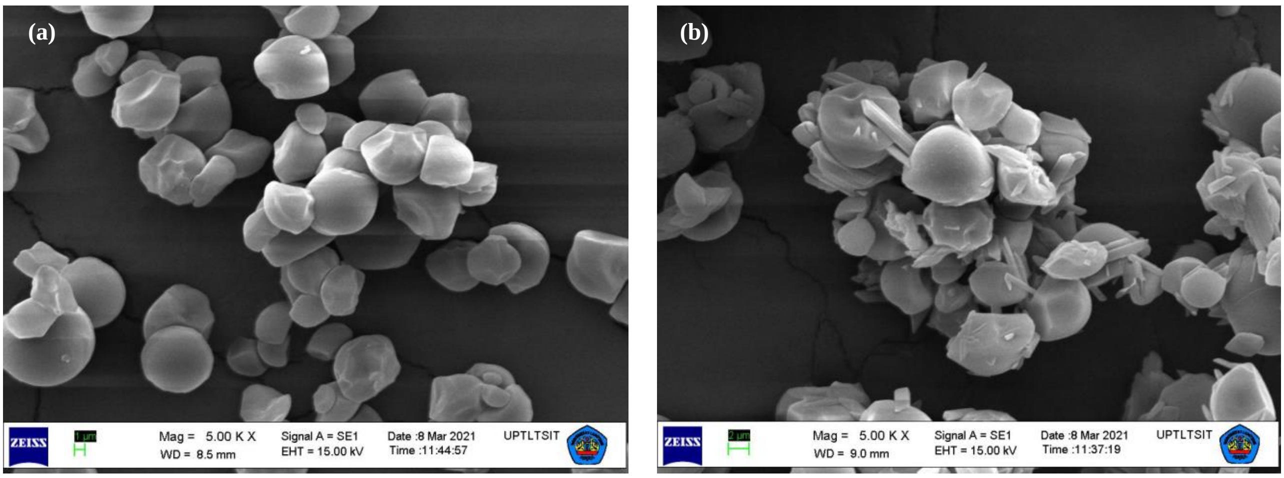

Figure 4. Morphology of breadfruit starch (a), starch succinate with time variation of 3.5 h at 105°C (b).

3.6 TGA analysis

Thermal properties describe the changes that occur during the heating of a starch until gelatinization occurs. These thermal properties can be analyzed using Thermogravimetric Analysis (TGA). The TGA pattern is presented in the form of the onset temperature (T0), peak temperature (Tp), and end temperature (Te). It also shows the changes in mass weight of the starch molecules that decrease as heat energy is applied. Based on Figure 5 for sample 0.5 h at 105°C, starch succinate has an onset temperature of 30°C, a peak temperature of 400°C, and an end temperature of 700°C. The decrease in temperature explains that starch succinate decomposes in three steps. At 150°C, the starch undergoes 10% decomposition, then decomposes at 400°C, resulting in a significant mass reduction of 82.9%, and finally loses mass at 650°C (94.7%). This aligns with the findings of Rudnik et al. (2005), which stated that starch decomposition begins at 150°C under both atmospheric and inert conditions. This indicates that the initiation of starch decomposition is non-oxidative. In addition to glass transition (TG) data, the DSC analysis includes a Differential Thermal Analysis (DTA) graph in Figure 5. DTA measures exothermic or endothermic changes with increasing temperature. The DTA curve shows an exothermic event, where a sharp peak appears at 420°C, corresponding to the complete breakdown of starch succinate (Wang et al., 2024).

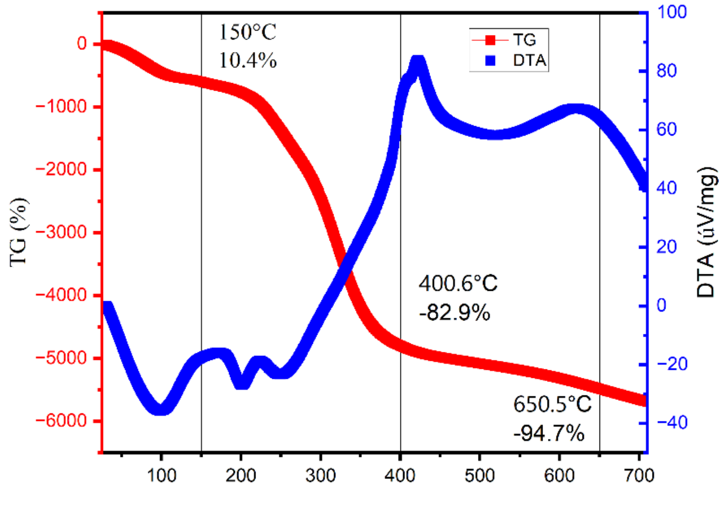

Figure 5. TGA graph

The data in the Table 2 shows the relationship among starch type, processing time, temperature, and degree of substitution (DS) in previous studies. Breadfruit starch processed for 2 h at 40°C had a DS value of 0.0241. Meanwhile, tannia (*Xanthosoma sagittifolium*) starch processed for 2 h at a higher temperature of 150°C had a greater degree of substitution (DS) of 0.0414. Cassava starch and Ginkgo starch, processed at a lower temperature (35°C) for 3 and 8 h respectively, showed smaller DS values of 0.008 and 0.006. Interestingly, Breadfruit starch in the designed study showed the highest DS value of 0.5293, which was much greater than the other samples. This indicates that the optimal combination of time and temperature can significantly increase the starch substitution rate. Based on the comparison of the degree of substitution (DS) value, the results are not worse than the previous research and are highly recommended for use in the industry (Altuna et al., 2018).

Table 2. Research results from previous studies

Sample	Time (h)	Temperature (°C)	Degree of substitution	Reference
Breadfruit starch	2	40	0.0241	Anwar et al. (2020b)
Tannia (*Xanthosoma sagittifolium*) starch	2	150	0.0414	Rosida et al. (2020)
Cassava starch	3	35	0.008	Zhang et al. (2017)
Ginkgo starch	8	35	0.006	Zheng et al. (2017)
Corn starch	3	40	0.086	Xie et al. (2024)
Potato starch	3	35	0.21	Chen et al. (2024)
Breadfruit Starch	3.5	105	0.5293	This study

4. CONCLUSION

This study successfully synthesized succinylated breadfruit starch through a succinylation reaction between native breadfruit starch and succinic acid using pyridine as a catalyst. The optimal reaction condition was achieved at 3.5 h and 105°C, yielding a degree of substitution (DS) value of 0.5293. The resulting modified starch appeared as a fine, homogeneous powder with a uniform texture. Fourier-transform infrared (FT-IR) spectroscopy confirmed the successful modification by detecting the characteristic ester carbonyl (C=O) absorption band at 1,692 cm^{-1}, indicating the formation of succinate ester groups. The succinylated starch synthesized under varying reaction times (3, 3.5, and 4 h) and temperatures (95, 105, and 115°C) exhibited increased swelling power and solubility compared to native starch. This enhancement is attributed to the molecular changes induced by the succinylation reaction and thermal treatment. Specifically, higher temperatures facilitate the depolymerization of amylose into shorter chains that are more soluble, while the increased proportion of amylopectin in the amorphous regions promotes greater water absorption and granule swelling. Thermogravimetric analysis (TGA) revealed that the starch underwent gelatinization in three distinct stages, with the most significant mass loss occurring at 400.6°C, corresponding to an 82.9% reduction in mass. Scanning Electron Microscopy (SEM) further demonstrated that succinylation altered the granule morphology; the starch granules became fragmented and exhibited surface disruption compared to the smooth, rounded granules of native breadfruit starch. These structural changes are consistent with the observed increases in swelling power and solubility, which were more pronounced with longer reaction times and higher temperatures. Overall, the results indicate that the succinylation reaction under optimized conditions effectively modifies the duration and temperature properties of breadfruit starch, enhancing its potential applicability in various industrial and food-related applications.

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AUTHORS CONTRIBUTION

Experimental run and Data Collection, Cut Fatimah Zuhra; Methodology, Validation, Supervision and Writing Original Draft Preparation, Andriayani.; Formal Analysis; Data Curation, Visualization, Writing - Review and Editing, Venny Angelina Siregar.

DECLARATION OF COMPETING INTEREST

The authors have no conflict of interest to declare.

REFERENCES

Adebowale KO, Olu-Owolabi BI, Olawumi EK, Lawal OS. Functional properties of native, physically and chemically modified breadfruit (*Artocarpus artilis*) starch. Industrial Crops and Products 2005;21:343-51.

Alam M, Pant K, Brar DS, Dar BN, Nanda V. Exploring the versatility of diverse hydrocolloids to transform techno-functional, rheological, and nutritional attributes of food fillings. Food Hydrocolloids 2024;146:Article No. 109275.

Altuna L, Herrera ML, Foresti ML. Synthesis and characterization of octenyl succinic anhydride modified starches for food applications. A review of recent literature. Food Hydrocolloids 2018;80:97-110.

Amari A, Elboughdiri N, Ghernaout D, Lajimi RH, Alshahrani AM, Tahoon MA, et al. Multifunctional crosslinked chitosan/ nitrogen-doped graphene quantum dot for wastewater treatment. Ain Shams Engineering Journal 2021;12(4):4007-14.

Anwar SH, Hasni D, Rohaya S, Antasari M, Winarti C. The role of breadfruit OSA starch and surfactant in stabilizing high-oil-load emulsions using high-pressure homogenization and low-frequency ultrasonication. Heliyon 2020a;6(7):e04341.

Anwar SH, Hasni D, Rohaya S, Antasari M, Winarti C. The role of breadfruit OSA starch and surfactant in stabilizing high-oil-load emulsions using high-pressure homogenization and low-frequency ultrasonication. Heliyon 2020b;6(7):e04341.

Bhosale R, Singhal R. Process optimization for the synthesis of octenyl succinyl derivative of waxy corn and amaranth starches. Carbohydrate Polymers 2006;66(4):521-7.

Błaszczak W, Fornal J, Kiseleva VI, Yuryev VP, Sergeev AI, Sadowska J. Effect of high pressure on thermal, structural and osmotic properties of waxy maize and Hylon VII starch blends. Carbohydrate Polymers 2007;68(3):387-96.

van der Burgt YEM, Bergsma J, Bleeker IP, Mijland PJHC, van der Kerk-van Hoof A, Kamerling JP, et al. Distribution of methyl substituents in amylose and amylopectin from methylated potato starches. Carbohydrate Research 2000; 325(3):183-91.

Chabib L, Nursal, Miskam M, Mohd Kaus NH, Shafie MH, Hamid M, et al. Optimization of g-C3N4 nanoparticles on structural, morphological, and optical properties as organic pollutants adsorbent in glycerin. Case Studies in Chemical and Environmental Engineering 2025;11:Article No. 101057.

Chen R, Ma Y, Chen Z, Wang Z, Chen J, Wang Y, et al. Fabrication and characterization of dual-functional porous starch with both emulsification and antioxidant properties. International Journal of Biological Macromolecules 2024;264(2):Article No. 130570.

Chen Y, Dai G, Gao Q. Preparation and properties of granular cold-water-soluble porous starch. International Journal of Biological Macromolecules 2020;144:656-62.

Chen Y, Hao Y, Ting K, Li Q, Gao Q. Preparation and emulsification properties of dialdehyde starch nanoparticles. Food Chemistry 2019;286:467-74.

Chi H, Xu K, Wu X, Chen Q, Xue D, Song C, et al. Effect of acetylation on the properties of corn starch. Food Chemistry 2008;106(3):923-8.

Deng Y, Jin Y, Luo Y, Zhong Y, Yue J, Song X, et al. Impact of continuous or cycle high hydrostatic pressure on the ultrastructure and digestibility of rice starch granules. Journal of Cereal Science 2014;60:302-10.

Dewi AMP, Santoso U, Pranoto Y, Marseno DW. Dual modification of sago starch via heat moisture treatment and octenyl succinylation to improve starch hydrophobicity. Polymers (Basel) 2022;14:Article No. 1086.

Fatimah Zuhra C, Ginting M, Masyita A, Fithri Az-zahra W. Carboxymethyl starch synthesis from breadfruit starch (*Artocarpus Communis*) through esterification reaction with monochloro acetate. Proceedings of the 1st International MIPAnet Conference on Science and Mathematics IMC-SciMath; Medan, Indonesia; 2022. p. 143-8.

Fatimah Zuhra C, Ginting M. Effect of essential oil of attarasa leaves (*Litsea Cubeba* Lour. Pers) on physico-mechanical and microstructural properties of breadfruit starch-alginate edible film. Malaysian Journal of Analytical Sciences 2013; 17(3):370-5.

Hamid M, Dayana I, Satria H, Siregar MF, Rianna M, Wijoyo H. Chitosan/CNDs coated Cu electrode surface has an electrical potential for electrical energy application. South Africa Journal Chemistry Enginering 2024a;50:445-50.

Hamid M, Humaidi S, Wijoyo H, Isnaeni I, Saragi IR, Simanjuntak C, et al. Solvothermal synthesized N-S doped carbon dots derived from cavendish banana peel (*Musa paradisiaca*) for detection of Fe(III) and Pb(II). Case Studies in Chemical and Environmental Engineering 2024b;10:Article No. 100832.

Hamid M, Kaus NHM, Humaidi S, Isnaeni I, Daulay A, Saragi IR. Activated carbon from biomass waste candlenut shells (*Aleurites moluccana*) doped ZIF-67/Fe_3O_4 as advanced materials for supercapacitor. Material Science Energy Technologies 2024c;7:381-90.

Hamid M, Humaidi S, Saragi IR, Simanjuntak C, Isnaeni I, Azizah, et al. The effectiveness of activated carbon from nutmeg shell in reducing ammonia (NH_3) levels in fish pond water. Carbon Trends 2024d;14:Article No. 100324.

Hao Y, Chen Y, Li Q, Gao Q. Synthesis, characterization and hydrophobicity of esterified waxy potato starch nanocrystals. Industrial Crops and Products 2019;130:111-7.

Haydersah J, Chevallier I, Rochette I, Mouquet-Rivier C, Picq C, Marianne-Pépin T, et al. Fermentation by amylolytic lactic acid bacteria and consequences for starch digestibility of plantain, breadfruit, and sweet potato flours. Journal Food Science 2012;77(8):466-72.

Huang TT, Zhou DN, Jin ZY, Xu XM, Chen HQ. Effect of repeated heat-moisture treatments on digestibility, physicochemical and structural propertiokres of sweet potato starch. Food Hydrocolloid 2016;54:202-10.

Humaidi S, Hamid M, Wijoyo H. Study and characterization of BaFe12O19/PVDF composites as electrode materials for supercapacitors. Biosensor Bioelectronic X 2024;19:Article No. 100507.

Luo H, Liu Q, Sun Z, Liang D, Zheng Y, Zhao L, et al. Removal of the out-shell for lotus root starch improved the effect of heat-moisture modification on multi-structure, physicochemical and digestibility properties. Food Hydrocolloid 2024;151:Article No. 109865.

Mei JQ, Zhou DN, Jin ZY, Xu XM, Chen HQ. Effects of citric acid esterification on digestibility, structural and physicochemical properties of cassava starch. Food Chemistry 2015;187:378-84.

Nasr JJM, Al-Shaalan NH, Shalan SM. Sustainable environment-friendly quantitative determination of three anti-hyperlipidemic statin drugs and ezetimibe in binary mixtures by first derivative Fourier transform infrared (FTIR) spectroscopy. Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy 2020;237:Article No. 118332.

Oderinde AA, Ibikunle AA, Bakre LG, Babarinde NAA. Modification of African breadfruit (*Treculia africana*, Decne) kernel starch: Physicochemical, morphological, pasting, and thermal properties. International Journal Biology Macromolecular 2020;153:79-87.

Ojogbo E, Ogunsona EO, Mekonnen TH. Chemical and physical modifications of starch for renewable polymeric materials. Materials Today Sustainability 2020;7-8:Article No. 100028.

Rosida, Finatsiyatull D, Nusandari R. Modification of native and hydrolyzed tannia (*Xanthosoma sagittifolium*) starch by succinic acid (succinylation). EurAsian Journal of BioSciences 2020:14(2):1-2

Rudnik E, Matuschek G, Milanov N, Kettrup A. Thermal properties of starch succinates. Thermochimica Acta 2005; 427(1-2):163-6.

Rumahorbo CGP, Ilyas S, Hutahaean S, Zuhra CF, Situmorang PC. The improvement of the physiological effects of Nanoherbal Sikkam leaves (*Bischofia javanica*). Rasayan Journal of Chemistry 2023;16:766-72.

Tan X, Li X, Chen L, Xie F, Li L, Huang J. Effect of heat-moisture treatment on multi-scale structures and physicochemical properties of breadfruit starch. Carbohydrat Polymer 2017;161:286-94.

Tan X, Zhang B, Chen L, Li X, Li L, Xie F. Effect of planetary ball-milling on multi-scale structures and pasting properties of waxy and high-amylose cornstarches. Innovative Food Science and Emerging Technologies 2015;30:198-207.

Volkert B, Lehmann A, Greco T, Nejad MH. A comparison of different synthesis routes for starch acetates and the resulting mechanical properties. Carbohydrat Polymer 2010;79(3):571-7.

Wang X, Chen L, Li X, Xie F, Liu H, Yu L. Thermal and rheological properties of breadfruit starch. Journal Food Science 2011;76(1):55-61.

Wang Yitong, Teng H, Bai S, Li C, Wang Ye, Ma L, et al. Pickering emulsion of camellia oil stabilized by Octenyl succinic acid starch: Interaction, lipid oxidation and digestibility. International Journal Biology Macromolecular 2024;279:Article No. 135108.

Wongsagonsup R, Varavinit S, BeMiller JN. Increasing slowly digestible starch content of normal and waxy maize starches and properties of starch products. Cereal Chemistry 2008;85(6):738-45.

Xie F, Liu X, Liu N, Feng X, He Z, Din Z ud, et al. Effect of degree of substitution of octenyl succinate on starch micelles for synthesis and stability of selenium nanoparticles: Towards selenium supplements. International Journal Biology Macromolecular 2024;280:Article No. 135586.

Zhang B, Mei JQ, Chen B, Chen HQ. Digestibility, physicochemical and structural properties of octenyl succinic anhydride-modified cassava starches with different degree of substitution. Food Chemistry 2017;229:136-41.

Zhang L, Xie W, Zhao X, Liu Y, Gao W. Study on the morphology, crystalline structure and thermal properties of yellow ginger starch acetates with different degrees of substitution. Thermochima Acta 2009;495(1-2):57-62.

Zheng Y, Hu L, Ding N, Liu P, Yao C, Zhang H. Physicochemical and structural characteristics of the octenyl succinic ester of ginkgo starch. International Journal Biology Macromolecularl 2017;94:566-70.

Zuhra CF, Ginting M, Azzahra WF, Hardiyanti R. Synthesis of bread fruit (*Artocarpus communis*)-based hydroxypropyl starch through etherification using propylene oxide. Rasayan Journal of Chemistry 2020;13:2445-54.

Zuhra CF, Sinaga MZE, Suharman, Situmorang PJ. Preparation of starch from breadfruit (*Artocarpus altilis*) with dual modification through oxidation and cross-linking methods. Applied Food Research 2025;5(1):Article No. 100787.