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ORIGINAL ARTICLE

Enhancing the functional characteristics of sago starch through dual chemical modification by hydroxypropylation and succinylation

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Abstract

Sago starch is a locally abundant starch indigenous of Indonesia. Despite its abundance, it is underutilized and restricted to food and packaging applications due to its limited functional characteristics. The value of native sago starch can be increased through modifications that improve its functionality, such as dual chemical modification. This sophisticated approach is more effective than single modification and makes the starch suitable for wider applications. Our study aimed to determine if dual chemical modification involving hydroxypropylation and succinylation would optimize the functional properties of sago starch. The sago starch was first modified by hydroxypropylation with 7 % (w/w) propylene oxide under alkaline conditions for 3 hours. This process resulted in hydroxypropylated starch with a substitution degree of 0.107. We then subjected the starch to succinylation using succinic anhydride at 1 % to 5 % of the starch weight in an alkaline solution for 2 hours. We achieved optimal functional characteristics of the dual-modified sago starch in the sample modified with 3 % (w/w) of succinic anhydride. The succinyl degree of substitution, water holding capacity, oil holding capacity, swelling power, and solubility of the dual-modified starch were 0.093, 4.16 $\mathrm{g\,g^{-1}}$, 7.20 $\mathrm{g\,g^{-1}}$, 34.25 $\mathrm{g\,g^{-1}}$, and 16.55 %, respectively. We conducted pasting properties analyses, infrared spectroscopy, and morphological structure analysis to determine the changes in the characteristics of the sago starch after hydroxypropylation and succinylation. The dual chemical modification successfully enhanced the functional characteristics of sago starch, particularly, its amphiphilic ability and swelling power. These results warrant further research and development in commercial applications.

Keywords: Amphiphilic Starches; Esterification; Etherification; Propylene Oxide; Succinic Anhydride.

1. Introduction

Sago palm is an abundant, indigenous starch source that is underutilized in Indonesia. Compared to other carbohydrate-producing crops, the sago palm is highly productive, capable of generating an annual dry starch yield ranging from 20 to 40 metric tons per hectare [1]. Indonesia possesses the largest sago plant holdings globally, encompassing approximately 1.128 million hectares, or 51 % of the world's sago area, distributed across 183 districts in 27 provinces [2]. The utilization of native sago starch (NS) has been primarily limited to processed and functional foods due to its inherent properties, which restrict its applicability to a broader range of uses.

NS has several limitations such as low solubility, susceptibility to retrogradation, inadequate thermal stability, insufficient thickening properties, limited shear resistance, and restricted shelf-life stability [3, 4]. These constraints can affect the function and performance of starch-based products, leading to lower overall efficacy and consumer acceptance. Previous studies have explored the potential of starch modifications, using various techniques to alter the properties of native starches with the aim of enhancing their functional characteristics. The development of dual-modified starches (DMS) offers a promising opportunity to significantly enhance efficacy, thereby expanding the range of applications.

Dual modification of starch can be achieved through physical, chemical, enzymatic, and heterogeneous methods [5]. Cross-linking, acid hydrolysis, oxidation, acetylation, grafting, esterification, and dual modification are the most common chemical modifications of starch structure in the industry due to their ease of control [6, 7]. This study examined two methods of NS modification, hydroxypropylation and succinylation. Hydroxypropylation is a starch modification technique that involves the substitution of hydroxyl groups with propylene oxide [8, 9, 10]. Succinylation is an esterification process that relies on succinic anhydride to introduce succinyl groups to the hydroxyl groups of starch [11, 12].

Previous studies have assessed the properties and applications of dual chemical modified starch. For example, a previous study evaluated dual modification through succinylation and crosslinking to enhance the physicochemical and functional characteristics of Mexican *O. tuberosa* tuber starch, particularly its emulsion stability and polymeric matrix properties [13]. Basilio-Cortés et al. Investigated the pasting and thermal profiles, as well as the morphology and functional group characteristics of dual-modified corn starch using hydrolysis and succinylation [14]. Another previous study performed a dual modification consisting of hydroxypropylation and alcoholic alkaline treatment to observe changes in the functional characteristics of sorghum and corn starch. The researchers studied characteristics such as swelling power, water holding capacity, rheology, infrared spectra, and shear stress [15]. A previous investigation combined hydroxypropylation and acid hydrolysis for the dual chemical modification of potato starch and determined the gel properties of the resulting product for applications such as encapsulation, hydrogels, and coatings [7]. Pakistani Basmati and Irri rice starches were modified using a combination of succinylation and crosslinking to investigate their functional, thermal, and morphological characteristics [16]. Mehfooz et al. Dual-modified barley starch through succinylation-crosslinking and oxidation-crosslinking to improve functional characteristics such as swelling, retrogradation, and thermal stability with implications for future food applications [17]. Shaikh et al. Performed succinylation of acid-thinned pearl millet starches to mitigate their high susceptibility to retrogradation [18]. Iftikhar et al. Conducted a dual modification consisting of acetylation and hydroxypropylation of rice starch to investigate if the modified product had enhanced characteristics that would warrant future food applications [19]. Regular and high-amylose maize starches were dually modified with acid hydrolysis and hydroxypropylation to understand the impact of the modifications on the amylose/amylopectin content [20].

However, there is a lack of research on the hydroxypropylation-succinylation of sago starch, which underscores the urgency for additional research to address this notable gap in the existing literature. In this study, we chose to explore hydroxypropylation and succinylation because no recent studies have examined these specific modification methods on sago starch. Hydroxypropylation and succinylation offer distinct potential advantages over prior methods. Further, hydroxypropilation, known for its ability to introduce hydrophilic groups [20, 21], and succinylation (esterification) have the potential to enhance the lipophilic characteristics of the starches [13, 14]. Thereby this dual modification has the potential to enhance the amphiphilic characteristics of starch that can improve its emulsification, encapsulation, and interfacial stabilization properties. While previous research on dual modified starches suggests promising outcomes, the specific impact of these modifications on sago starch remains to be fully elucidated.

This study aimed to evaluate the effects of hydroxypropylation and succinylation on the functional properties of sago starch. It provided a comprehensive analysis of the observed changes to better understand the potential benefits and applications of these modification techniques. We specifically studied how varying levels of succinylation after hydroxypropylation affected sago starch, aiming to identify the conditions that lead to optimal functional properties.

2. Materials and Methods

2.1. Materials

Commercial NS was purchased from PT Bangka Asindo Agri, Indonesia. The NS was dried to maintain a moisture content of up to 12 % w/w for sample preparation. All chemicals, including propylene oxide, succinic anhydride, sodium sulfate, sodium hydroxide, propylene glycol, and hydrochloric acid 32 % aqueous solution were of analytical grade and ordered from Merck (Darmstadt, Germany). Distilled water was utilized for all experiments.

2.2. Hydroxypropylated sago starch

A homogeneous mixture was prepared by combining 250 g of NS with 395 g of aqueous sodium sulfate (10 % w/w) in a flask, resulting in a starch suspension (40 % w/w) that weighed 645 g. The pH of the suspension was adjusted to 12 by adding a 1M NaOH. Next, 18 mL of an aqueous propylene oxide solution (7 % w/w) were added to the suspension. The mixture was agitated for 30 minutes in a covered flask. The hydroxypropylation process involved agitating the suspension at 150 rpm for 3 hours in a water bath preheated to 40 °C. The process was terminated by cooling the suspension and neutralizing it with 0.5M HCl until the pH stabilized within a range of 6.5-7. The suspension was then filtered and washed with water multiple times. The resulting material, referred to as “the cake,” was dried overnight in an oven at 50 °C until its moisture content was reduced to less than 12 % w/w. The dried cake of hydroxypropylated sago starch (HPS) was then milled to produce a powder with a mesh size of 80.

2.3. Succinylation of hydroxypropylated sago starch

A sample consisting of 150 g of HPS and water in a 1:3 mass ratio was prepared and replicated five times. The suspension was stirred magnetically for 1 hour before adding 1M NaOH to adjust the pH to 9. Succinic anhydride was added to each sample at weights of 1.5 g, 3 g, 4.5 g, 6 g, and 7.5 g (1 %, 2 %, 3 %, 4 %, 5 % w/w based on the weight of HPS). The pH of the suspension was readjusted to a range of 8-9 with 1M NaOH. The suspension was then subjected to gentle stirring for 2 h at room temperature. The procedure was completed by neutralizing the suspensions using a 0.5M HCl solution until the pH suspension was 6.5-7. The suspension was then filtered, and the resulting cake was washed three times with distilled water before being dried in an oven at 50 °C overnight until its moisture content reached a maximum of 12 % w/w. The dried cakes (labeled as DMS1, DMS2, DMS3, DMS4, and DMS5) were ground to a mesh size of 80.

2.4. Degree of substitution of hydroxypropylation (DSH)

All samples, weighing 0.1 g, were mixed with 25 mL of 1N H_2SO_4 solution in a 100-mL volumetric flask. The flasks were heated in boiling water until the samples were completely dissolved and then cooled with water. The samples were then diluted and scaled up to a final volume of 100 mL. The resulting samples were diluted and scaled up to a final volume of 100 mL. Additional dilution was necessary to ensure that the concentration of hydroxypropyl groups was below 0.04 mg mL^{-1}. A total of 10 mL of sample and 8 mL of H_2SO_4 (98 %, w/w) were added to a test tube that was later heated in boiling water for 3 min and then chilled in an ice bath. A total of 0.6 mL of the Ninhydrin reagent (3 % w/w ninhydrin solution in 5 % w/w aqueous sodium bisulfite) was gently poured along the wall of the tube. After thorough mixing, the samples were left at room temperature for 100 minutes. The samples were then diluted with concentrated sulfuric acid to a volume of 25 mL, and the tubes were repeatedly inverted (not shaken). The samples were placed in 1 cm cells for 5 minutes before being measured using a UV-Vis spectrophotometer at 590 nm (Merck, Spectroquant Pharo 300, Germany). Aliquots of standard solutions containing 10, 20, 30, 40, and 50 g of propylene glycol per mL were used to create the calibration curve [22]. The percentage of hydroxypropyl groups (HP%) and the degree of substitution of hydroxypropyl groups (DSH) were determined using Eq. 1 and Eq. 2, respectively:

$$\text{HP}\ \% = \frac{\text{PG} \times 0.7763 \times 10 \times F}{W}, \tag{1}$$

$$\text{DSH} = \frac{162 \times \text{HP\%}}{5800 - (58 \times \text{HP\%})}, \tag{2}$$

where PG is the amount of Propylene Glycol ($\mu\text{g mL}^{-1}$) in the sample obtained from the calibration curve, F is the dilution factor (if further dilution was required), 0.7763 is the proportion factor of hydroxypropyl groups to milligrams of propylene glycol, and W is the sample weight (mg).

2.5. Degree of substitution of succinylation (DSS)

The DSS value was determined using saponification according to the respective method [23]. Briefly, 25 mL of aqueous ethanol (75 %, w/w) were added to a 100-mL cylindrical beaker. Followed by 0.5 g of sample and by 20 mL of 0.5 M aqueous sodium hydroxide solution. The solution was kept at room temperature with intermittent rotation for 72 h. Back titration with a volume of 0.5 M hydrochloric acid solution was used to neutralize the alkali remnants. The percentage of succinyl groups (SUC%) and the degree of substitution of succinylation groups (DSS) were determined using Eq. 3 and Eq. 4:

$$\text{SUC}\ \% = \frac{(B - S) \times 0.1 \times M \times 100}{W}, \tag{3}$$

$$\text{DSS} = \frac{162 \times \text{SUC\%}}{10000 - (99 \times \text{SUC\%})}, \tag{4}$$

where B is the blank sample titer (mL), S is the sample titer (mL), M is the acid molarity (M), and W is the sample weight (g).

2.6. Water holding capacity (WHC)

The method proposed by [24] was carried out with a change in centrifugation speed and time. The sample, weighing 0.2 g on a dry basis, was diluted in 4 mL of distilled water. The suspension was left at room temperature for 10 minutes before centrifugation (Hitachi 05P-21, Japan) at 900 x g for 25 min. The sediment was weighed after decanting the supernatant. The water holding capacity (WHC) was determined using Eq. 5:

$$\text{WHC} = \frac{W_1}{W_0}, \tag{5}$$

where W_1 is the weight of the sediment (g) and W_0 is the initial weight of the sample (g).

2.7. Oil holding capacity (OHC)

This techinque was also conducted using the method described by [24] with minor adjustments regarding the sample and vegetable oil mass ratio, as well as the speed and duration of the centrifugation process. The sample, weighing 0.5 g on a dry basis, was diluted in 6 mL of vegetable oil. The solution was then mixed vigorously for 1 min with a vortex mixer and left at room temperature for 30 min before centrifugation (Hitachi 05P-21, Japan) at 900 x g for 40 min. The sediment was weighed after decanting the supernatant. The oil holding capacity (OHC) was determined using Eq. 6:

$$\text{OHC} = \frac{W_1}{W_0}, \tag{6}$$

where W_1 is the weight of the sediment (g) and W_0 is the initial weight of the sample (g).

2.8. Swelling power

The swelling power analysis was performed as described by [10]. A starch sample weighing 0.1 g on a dry basis was combined with 10 mL of distilled water, thoroughly mixed in a 50 mL centrifuge tube, and heated at 70 °C for 30 min in a shaking water bath before centrifugation (Hitachi 05P-21, Japan) at 900× g for 1 h. The sediment was weighed after decanting the supernatant. The swelling power was determined using Eq. 7:

$$\text{Swelling Power} = \frac{W_1}{W_0}, \tag{7}$$

where W_1 is the weight of the sediment (g) and W_0 is the initial weight of the sample (g).

2.9. Solubility

The method from [25] was employed with few modifications regarding cooling temperature. A starch sample weighing 0.1 g on a dry basis was mixed with 9.9 mL of distilled water and heated at 90 °C for 1 h in a water bath. Then, the sample was quickly cooled in an ice water bath for 1 min, followed by additional cooling at room temperature for 5 min. The sample was then centrifuged (Hitachi 05P-21, Japan) at 900× g for 30 min. The supernatant was transferred to a pre-weighed Petri dish, dried in an oven at 105 °C overnight, then cooled to room temperature in a desiccator before reweighing. Solubility was determined using Eq. 8:

$$S = \frac{W_s}{W_0} \times 100, \tag{8}$$

where S is the sample solubility (%), W_s is the weight of the dried supernatant (g), and W_0 is the initial weight of the dry starch (g).

2.10. Gelation characteristics by least gel concentration method

The gelation characteristics were investigated using the technique proposed by [26] with modifications regarding the variation in concentration of the suspension. Sample suspensions at concentrations of 2 %, 4 %, 6 %, 8 %, and 10 % (w/v) in 10 mL of distilled water were boiled for 1 h and promptly cooled in ice-cold water. The suspensions were further cooled at 4 °C for 2 h. The least gel concentration indicated the point where gelation occurred effectively was determined as the lowest concentration at which the sample did not slide down or slip when the test tube was reversed.

2.11. Fourier transform infrared spectroscopy

Fourier transform infrared (FTIR) spectroscopy (Bruker Alpha II, Germany) was used to determine the chemical bonds present in sample molecules by assessing the intensity of peak absorbance of the functional groups. A total of 0.1-0.5 g of sample powder was prepared and placed on the diamond ATR crystal of the FTIR system using a stainless-steel laboratory scraper or a similar instrument. Pressure was applied to the material using a pressure anvil. The sample was analyzed using the FTIR system software over the range of 400-4000 cm^{-1} to characterize the molecular absorption of the sample.

2.12. Pasting properties

An amylograph was used to characterize the pasting characteristics of the starch samples (Brabender Amylograph-E, Germany). The sample (30 g, dry basis) was thoroughly suspended in 470 g of distilled water before being transferred to a Brabender bowl. The equipment was programmed for a heating and cooling cycle, starting with heating from 30 °C to 93 °C, holding at 93 °C for 20 min, cooling to 50 °C, and holding at 50 °C for another 20 min. The experimental protocol involved maintaining a constant stirrer speed of 75 rpm, applying a temperature ramp of 1.5 °C/min, and exerting a force of 700 cm-g (equivalent to 1,000 Brabender Units). Pasting characteristics such as pasting temperature (PT), peak viscosity (PV), final viscosity (FV), setback viscosity (SBV), and breakdown viscosity (BDV) were determined using the Brabender system software in Brabender Units (BU).

2.13. Scanning electron microscopy

The morphological structure of the sample was examined using an environmental scanning electron microscope (Thermo Fisher Scientific Quattro S, USA). The sample was mounted on a standard aluminum pin stub measuring 12.5 mm in width, which was then placed on the designated mount and inserted into the chamber of the equipment. The sample was scanned at an acceleration voltage of 20 kV and a magnification of 1500×.

2.14. Statistical analysis

All numerical results are presented as mean values of triplicates ± standard deviation (SD). The data were analyzed using a one-way analysis of variance (ANOVA) and Tukey test with Minitab 21.3 (Pennsylvania, USA) to assess significant differences between the mean values (p-value < 0.05). Different superscript letters (a, b, c, d, e, f, and g) indicate significant differences.

3. Results and Discussion

3.1. Degree of Substitution of Modified Sago Starch

The modification of starch and its resulting degree of substitution are critical factors that determine the properties and characteristics of the final material. The degree of substitution value represents the amount of modifying agent added per unit of glucose in the starch. The reaction was conducted with adjustments made to the amount of reagent, reaction time, temperature, and pH. Hydroxypropylation, the initial modification, is a chemical process where a propyl group from propylene oxide is introduced into the hydroxyl groups of the starch molecule, resulting in hydroxypropyl groups. After the initial phase of modification was completed, the DSH value was 0.107 ± 0.001. This value falls within the suitable range for the use of hydroxypropylated starch, particularly in food applications. The hydroxypropylated starch used in food applications adheres to the United States Food and Drug Administration's (USFDA) recommended limit, which should not be above 0.2 [27].

The second modification step consisted of introducing succinyl groups onto the starch granules through an esterification reaction, a process known as succinylation. This reaction involved substituting hydroxyl groups in the starch granules with succinyl groups derived from succinic anhydride. This modification aimed to enhance the properties of hydroxypropylated starch, thereby broadening its potential for use in various industrial applications. **Table 1** provides a detailed overview of the DS values of the dual-modified sago starch (DMS).

The DS results ranged from 0.033 to 0.096, with the highest value obtained with DMS3. The incorporation of succinyl groups increased gradually until the concentration of succinic anhydride reached 3 % w/w (DMS3). At higher concentrations, the DS value started to decrease. According to USFDA regulations, (21 CFR 175.892), succinic anhydride is generally recognized as safe (GRAS) for miscellaneous and general purposes, with no additional limitations on its usage beyond adherence to good manufacturing practices [28]. However, the percentage used in this study remained below the permissible upper limit of 4 % for succinic anhydride in starch-based food products as regulated by the USFDA [29].

The decrease in DS values observed for DMS4 and DMS5 can be attributed to the higher concentrations of succinic anhydride used and the advanced stage of modification within the starch molecules, which likely resulted from the dual modification process. Furthermore, excessive succinic anhydride, especially following propylene oxide treatment, could hinder the efficiency of succinylation through mechanisms such as limiting accessibility to reactive sites on the starch, promoting competing side reactions, inducing hydrolysis of ester linkages, and forming higher molecular weight aggregates that are less reactive towards starch molecules. Previous studies have also reported a decrease in DS values when succinic anhydride levels reach saturation, typically between 2-4 % w/w based on the weight of the native starch [30, 31].

3.2. Water and oil holding capacity

The WHC values of DMS showed a slight increase from 3.61 to 4.16 ($\mathrm{g\,g^{-1}}$) with increasing succinic anhydride concentrations (DMS1 to DMS3), before decreasing to 3.73 ($\mathrm{g\,g^{-1}}$) for DMS5 (Table 1). Although these results did not reach statistical significance, we observed a trend of increasing WHC values at higher concentrations of succinic anhydride, followed by a decrease at concentrations above 3 % (w/w). Moreover, the DMSs exhibited significantly higher WHC values compared to the HPS and NS. Hydroxypropylation and succinylation enhanced the hydrophilic nature of sago starch by altering its structure. Both modifications are attributed to the introduction of hydrophilic groups, which induce steric hindrance that facilitates the hydration of starch granules [31]. The decrease in WHC values observed in DMS4 and DMS5 may be attributed to excessive starch modifications, which hindered the ability of the starch granule to absorb water effectively.

Table 1. Degree of substitution, water holding capacity, and oil holding capacity of the native and modified sago starches.

No.	Modified Sago Starch	DMS Value	Water holding capacity ($g\,g^{-1}$)	Oil holding capacity ($g\,g^{-1}$)
1	DMS1	0.033^{d}	3.61 ± 0.06^{cd}	5.57 ± 0.07^{c}
2	DMS2	0.065 ± 0.001^{c}	3.67 ± 0.03^{cd}	6.47 ± 0.05^{b}
3	DMS3	0.093 ± 0.001^{a}	4.16 ± 0.07^{a}	7.20 ± 0.03^{a}
4	DMS4	0.087 ± 0.001^{ab}	3.88 ± 0.03^{b}	7.08 ± 0.09^{a}
5	DMS5	0.072 ± 0.001^{bc}	3.73 ± 0.08^{bc}	6.63 ± 0.08^{b}
6	HPS	-	3.57 ± 0.08^{d}	3.09 ± 0.04^{d}
7	NS	-	2.37 ± 0.04^{e}	2.38 ± 0.04^{e}

The variations in OHC values of dual-modified starches with varying concentrations of succinic anhydride are presented in Table 1. Although the difference between samples was not statistically significant, we observed a slight improvement between DMS1 and DMS3 [from 5.57 to 7.20 ($g\,g^{-1}$)] with increasing succinic anhydride concentrations up to 3% (w/w), followed by a decrease to 6.63 $g\,g^{-1}$ (DMS5). Esterification (via succinylation) is employed for lipophilic substitution, involving nucleophilic attack in aqueous solutions under mild alkaline conditions. This process predominantly occurs on the surface and amorphous regions of the hydrophilic starch granules, leading to the addition of lipophilic groups and the development of amphiphilic features [13]. There was no significant difference between dual modified starches, but the OHC value of DMS5 was slightly higher than that of DMS2, DMS3, and DMS4. Moreover, the slight decrease in OHC values observed from DMS4 to DMS5 could be attributed to the excessive addition of succinic anhydride, which modified the starch and consequently hindered its ability to effectively absorb oil.

3.3. Swelling power and solubility

The swelling power values of the modified starches are shown in **Table 2**. Swelling power is a fundamental starch property that describes the ability to absorb water and increase in size. The swelling power of HPS (23.35 $g\,g^{-1}$) was significantly higher than that of NS (16.24 $g\,g^{-1}$), and the swelling power of the DMS samples was even higher. The swelling power value increased sharply from 27.02 $g\,g^{-1}$ for DMS1 to 34.25 $g\,g^{-1}$ for DMS3, then decreased to 33.54 $g\,g^{-1}$ for DMS 4 and 30.79 $g\,g^{-1}$ DMS5. DMS5 had a slightly higher swelling power than DMS2, but the differences were not statistically significant. Hydroxypropylation and succinylation alter starch molecules in different ways. Hydroxypropylation introduces hydrophilic groups, increasing the starch's hydration capacity, whereas succinylation introduces anionic groups, enhancing the starch's ability to attract water. According to núñez-bretón et al [13], succinylation enhances the swelling capacity of modified starch by replacing hydroxyl groups with larger succinyl groups. This creates steric hindrance, which facilitates water penetration into the starch granules. However, in samples DMS4 and DMS5, and excess of succinyl groups may have rigidified the molecular structures of starch molecules, potentially reducing their ability to expand and disperse in water.

Solubility refers to the ability of starch molecules to dissolve in water or other solvents. The solubility of the DMS samples differed significantly from that of HPS and NS and HPS exhibited the highest solubility (29.01 %). Hydroxypropylation of starch enhances its solubility and swelling power by disrupting hydrogen bonds within starch chains, introducing hydrophilic bulky groups to the starch backbone, and improving water accessibility [20, 32]. The solubility of dual modified starches decreased following succinylation. Despite having lower values compared to HPS, the solubility of DMSs increased from 12.36 % to 16.55 % as the succinic anhydride concentration increased to 3 % w/w (DMS1 to DMS3), before declining to 14.14 % for DMS5. The decrease in solubility could have been caused by excessive succinylation, which may induce structural changes and the formation of aggregates within the starch molecules. These changes could ultimately diminish solubility by impairing the interaction with water. The low solubility observed after succinylation of sago starch was reported in a previous study by Marta *et al* [33]. This phenomenon was attributed to the formation of strong bonds between amylose and succinyl groups, which trapped water molecules within the starch molecules and hindered the release of amylose molecules. Calderón-Castro [11] also noted that succinylation of corn starch via an extrusion process increased the hydrophobicity of starch molecules, which are commonly hydrophilic.

Table 2. Swelling power and solubility of the native and modified sago starches.

No.	Modified Sago Starch	Swelling Power ($g\,g^{-1}$)	Solubility (%)
1	DMS1	27.02 ± 0.09^{d}	12.36 ± 0.05^{f}
2	DMS2	30.32 ± 0.07^{c}	13.55 ± 0.08^{e}
3	DMS3	34.25 ± 0.13^{a}	16.55 ± 0.07^{b}
4	DMS4	33.54 ± 0.18^{b}	16.05 ± 0.08^{c}
5	DMS5	30.79 ± 0.15^{c}	14.14 ± 0.09^{d}
6	HPS	23.35 ± 0.10^{e}	29.01 ± 0.08^{a}
7	NS	16.24 ± 0.36^{f}	11.31 ± 0.03^{g}

3.4. Least gel concentration

The concept of Least Gelation Concentration (LGC) refers to the minimum concentration of a gelling agent required to form a gel that maintains its form. The modifications assessed in this study reduced the gel-forming ability of the starches. HPS, DMS1, DMS2, DMS3, DMS4, and DMS5 required 8 % w/v of the gelling agent to form a firm gel, which is 2 % higher than the concentration required for NS (**Table 3**). The lower LGC could be attributed to the involvement of amylose macromolecules in retrogradation, potentially enhancing the gel's strength [34], which correlates with the lowest SBV values in the NS pasting profile (**Table 4**).

Table 3. Least gel concentration of the native and modified sago starches.

No.	Modified Sago Starch	2 % (w/v)	4 % (w/v)	6 % (w/v)	8 % (w/v)	10 % (w/v)	LGC (%)
1	DMS1	Viscous	Viscous	Viscous	Firm Gel	Firm Gel	8
2	DMS2	Viscous	Viscous	Viscous	Firm Gel	Firm Gel	8
3	DMS3	Viscous	Viscous	Viscous	Firm Gel	Hard Gel	8
4	DMS4	Viscous	Viscous	Viscous	Firm Gel	Hard Gel	8
5	DMS5	Viscous	Viscous	Viscous	Firm Gel	Firm Gel	8
6	HPS	Viscous	Viscous	Viscous	Firm Gel	Firm Gel	8
7	NS	Viscous	Viscous	Firm Gel	Firm Gel	Hard Gel	6

The amorphous regions of starches are crucial for swelling, hydration, and ultimately gel formation. Furthermore, the gel strength depends on the intragranular binding forces within the swollen starch granules, which maintain the structural integrity of starch gels. For stronger gels to form, the functional groups that contribute to intragranular binding forces, such as hydrogen bonds, must remain intact. The substitution of succinyl groups on starch molecules disrupts intragranular force interactions by replacing OH groups on the glucose units, resulting in weaker gels than those of native starch. The incorporation of succinyl groups likely contributed to reduced gel cohesiveness and decreased gel strength.

3.5. Fourier transform infrared spectroscopy

FTIR determines the molecular composition of a sample by measuring the amount of infrared light absorbed or transmitted. **Figure 1(a)** illustrates the FT-IR spectra of both native and modified starches. The hydroxyl groups in the native starch were effectively intercepted by hydroxypropyl groups, resulting in a slight increase in peak intensities around 2975 cm^{-1}. Notably, the hydroxyl groups originally positioned at 2970 cm^{-1} in the NS were shifted (**Figure 1(b)**). This finding was similar to that of a previous study on hydroxypropylated sago starch, where a small peak of hydroxypropyl groups was observed at 2974 cm^{-1} [10]. Furthermore, two distinctive signals highlighted the substitution of hydroxyl groups with succinyl groups: the carboxyl groups peak and the stretching vibration of the ester groups [13, 14, 35–37]. We confirmed that esterification by succinylation occurred due to the emergence of new peaks and higher intensities at 1562 cm^{-1} (carboxyl groups) and 1725 cm^{-1} (ester carbonyl groups) (**Figure 1(c)**). A comparable study of esterification by acetylation for glutinous rice starch observed a peak in the carbonyl groups at 1728 cm^{-1} [38], which is nearly identical to the succinylation spectra observed in this study. The observed variations in peak levels within the aforementioned region indicated differences in the concentration of the reagents. DMS3 presented the highest peak of absorbance for carboxyl groups and a slightly higher peak for carbonyl groups, nearly identical to that of DMS4. The absorbance peaks for DMS3 and DMS4 were 0.01774 and 0.01768, respectively.

3.6. Pasting properties

When using starch in various processes, it is crucial to study the behavior of its viscosity. The alteration in viscosity of starch suspensions due to temperature fluctuations were assessed, as shown in Table 4. Despite the pasting profiles of the dual-modified starches, HPS and NS did not show statistically significant differences. The modified starches exhibited lower pasting temperatures (PT) and higher peak viscosities (PV) than NS. This is because the hydrophilic hydroxypropyl and succinyl groups degraded the granular starch structures during the modification process, enabling water molecules to permeate at lower temperatures. DMS3 presented the highest PV (1496.67 BU), standing out from the other samples due to extensive modifications that significantly enhanced the starch's swelling and water absorption capacity. Moreover, DMS3 yielded the lowest PT at 65.73 °C. The PT value subsequently increased for DMS4 and DMS5. The decrease in PV and increase in PT observed at higher succinylation concentrations (DMS4 and DMS5) could be attributed to the excessive concentration of succinic acid used during the modification.

The breakdown viscosity (BDV) values of the dual-modified starches (DMS1-DMS5) exceeded those of HPS and NS. However, the BDV value of HPS was lower than that of NS, suggesting that hydroxypropylated starch exhibited greater resistance to severe processing conditions and high-temperature shearing. DMS3 presented the maximum BDV value (670.67 BU), which then decreased as the succinic anhydride concentration increased. This decrease resulted from the reduced rigidity of DMS4 and DMS5, caused by excessive modification that damaged the

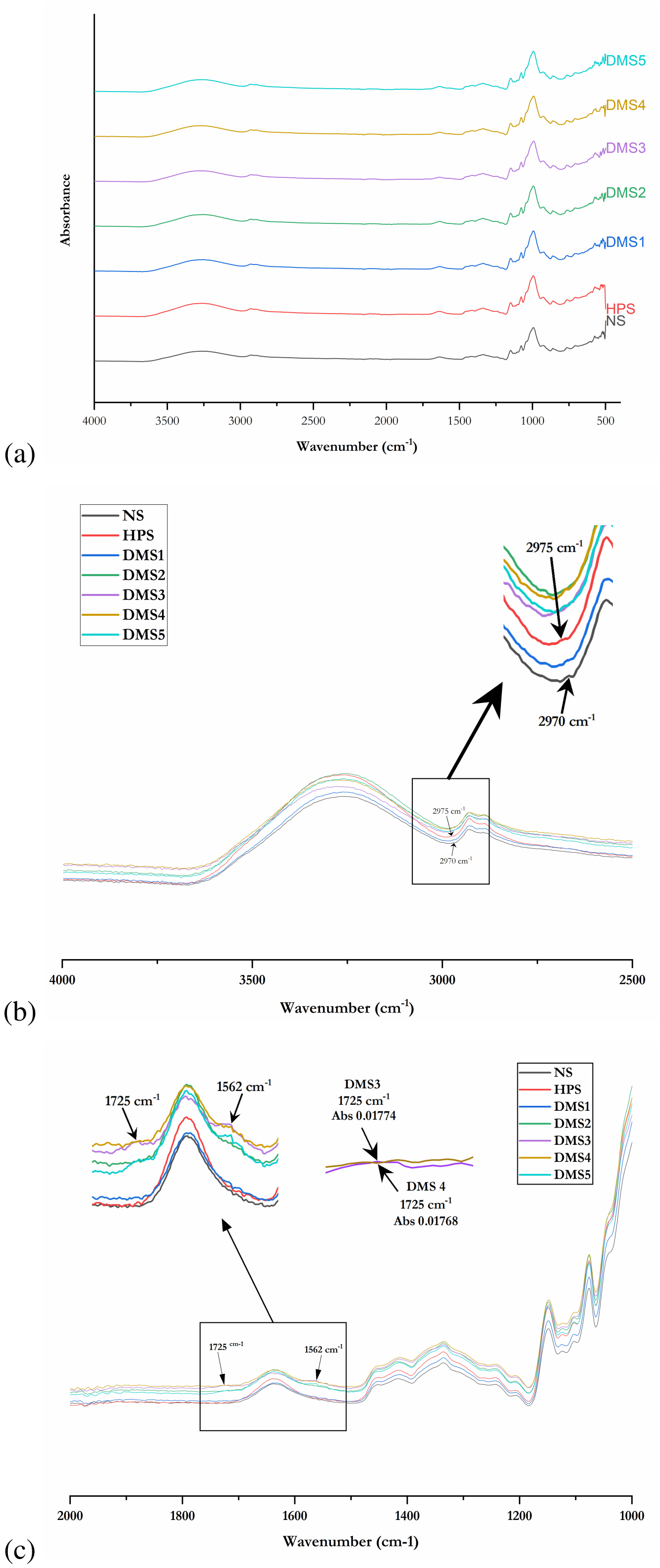

Figure 1. FTIR spectra of native and modified sago starches in the range of 4000-400 cm^{-1} (a), with a small peak for HPS at 2975 cm^{-1} (b), and succinyl group peaks at 1562 and 1725 cm^{-1} (c).

granule structures. Moreover, the extensive alterations made the succinylated granules more elastic, ultimately rendering them more fragile and leading to disintegration due to the shearing effect [39].

The setback viscosity (SBV) reflects the stability of starch gel and its tendency for retrogradation, as it quantifies the rise in viscosity when a starch gel is cooled and subsequently reheated. A lower SBV value is associated with stability in the starch gel and a reduced propensity to retrogradation. The SBV value of all modified starches exceeded that of the NS, suggesting that the modifications resulted in an increased propensity to retrogradation. DMS3 presented the highest SBV value (341 BU), which subsequently decreased with further increases in succinic anhydride concentrations. This phenomenon could be attributed to an excessive dosage of the reagent, which increased the tendency for intermolecular reassociation between modified starch molecules.

Table 4. Comparison of pasting characteristics between native and modified sago starches.

No.	Modified Sago Starch	Pasting Temperature (°C)	Peak Viscosity (BU)	Final Viscosity (BU)	Breakdown Viscosity (BU)	Setback Viscosity (BU)
1	DMS1	68.00 ± 0.66^{abc}	1177.00 ± 13.08^{b}	997.00 ± 19.52^{b}	429.00 ± 33.00^{cd}	286.67 ± 0.58^{b}
2	DMS2	68.30 ± 0.36^{ab}	1141.00 ± 22.10^{b}	891.00 ± 14.18^{c}	455.33 ± 19.76^{bc}	266.67 ± 4.04^{b}
3	DMS3	65.73 ± 1.26^{c}	1496.67 ± 37.29^{a}	1187.00 ± 23.58^{a}	670.67 ± 24.09^{a}	341.00 ± 12.77^{a}
4	DMS4	67.43 ± 1.57^{bc}	1170.33 ± 26.01^{b}	927.67 ± 29.01^{c}	525.67 ± 27.47^{b}	276.67 ± 11.93^{b}
5	DMS5	67.50 ± 0.50^{bc}	1176.33 ± 34.85^{b}	1006.00 ± 33.45^{b}	368.00 ± 22.65^{d}	276.67 ± 2.31^{b}
6	HPS	69.87 ± 0.23^{ab}	805.00 ± 28.83^{c}	717.67 ± 21.22^{d}	291.67 ± 28.75^{e}	222.70 ± 22.37^{c}
7	NS	70.47 ± 1.00^{a}	634.67 ± 12.90^{d}	454.33 ± 12.34^{e}	358.67 ± 26.63^{de}	188.67 ± 5.77^{d}

3.7. Scanning electron microscopy (SEM) analysis

The morphological alterations of the modified samples were examined and presented in **Figure 2**. The NS granules exhibited smooth and oval forms, consistent with the findings of a previous study [33]. The hydroxypropylation process deformed the NS granules, causing them to rupture and tear, resulting in smaller shapes. The granules displayed a higher degree of deformation and cracking as the concentration of succinic anhydride increased. However, despite the deformation, the granular shapes retained their basic structure and did not transform into entirely new forms. A similar process was reported in a previous study, where succinylation did not entirely eliminate the granular form of corn starch granules [23].

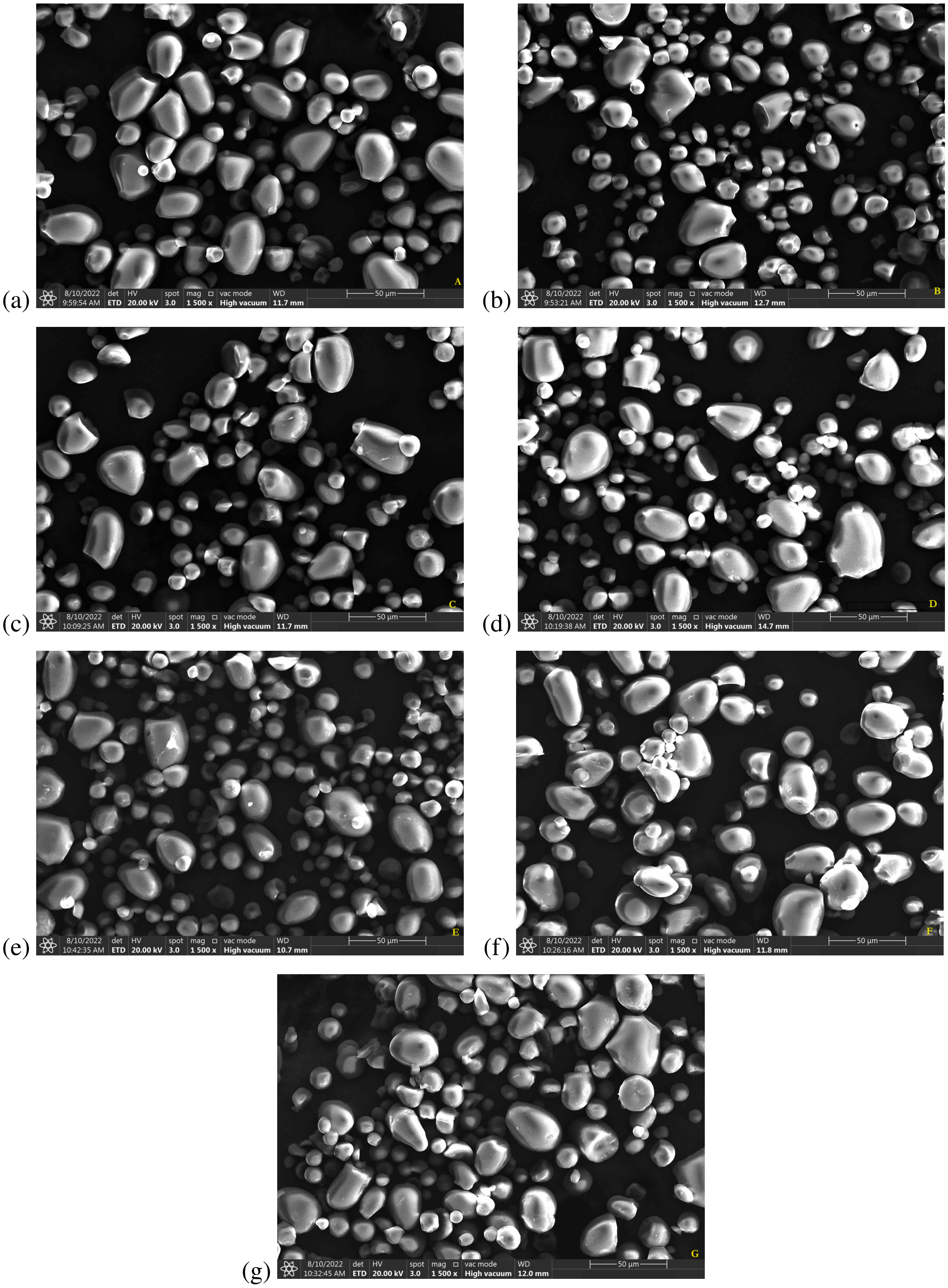

Figure 2. Morphological alterations in dual-modified sago starch: (a) NS; (b) HPS; (c) DMS1; (d) DMS2; (e) DMS3; (f) DMS4; (g) DMS5.

4. Conclusions

This study examined the dual modification of sago starch through hydroxypropylation and succinylation. Succinylation significantly improved the functional characteristics of sago starch, including starch viscosity, swelling power, WHC, and OHC. The dual-modified starches were less soluble than the HPS and NS because the succinyl groups strongly bonded with the starch molecules, preventing the release of amylose. The extent of improvement depended on the concentration of the reagent used. According to our results, the concentration of succinic anhydride that achieved the best functional characteristics was 3 % w/w. Excessive succinylation led to starch degradation and decreased performance, and it also violated FDA regulations on the maximum allowable reagent content in specific starch products. The results of our study warrant further research to better understand the applications of DMS in specific industrial settings. Potential applications include adsorbents, functional foods, and the paper industry, where significant enhancements in functional characteristics have been observed.

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6. Conflicts of interest

The authors declare no conflict of interest.

References

[1] Bintoro MH, Nurulhaq MI, Pratama AJ, Ahmad F, Ayulia L. Growing area of sago palm and its environment, *Sago Palm: Multiple Contributions to Food Security and Sustainable Livelihoods*, 17–29, 2018.

doi: 10.1007/978-981-10-5269-9_2

[2] Yusuf MA, Romli M, Suprihatin, Wiloso EI. Carbon footprint of semi-mechanical sago starch production, *Journal of Ecological Engineering*, 20(11):159-166, 2019.

doi: 10.12911/22998993/110813

[3] Ayuningtyas LP, Benita AM, Triastuti D. Functional properties of hydrothermally modified lesser yam (Dioscorea esculenta) starch, *IOP Conference Series: Earth and Environmental Science,* 746:012007, 2021.

doi: 10.1088/1755-1315/746/1/012007

[4] Shahbazi M, Majzoobi M, Farahnaky A. Impact of shear force on functional properties of native starch and resulting gel and film, *Journal of Food Engineering*, 223:10–21, 2018.

doi: 10.1016/j.jfoodeng.2017.11.033

[5] Ashogbon AO. Dual modification of various starches: Synthesis, properties and applications, *Food Chemistry*, 342:128325, 2021.

doi: 10.1016/j.foodchem.2020.128325

[6] Haq F, Yu H, Wang L, Teng L, Haroon M, Khan RU, Mehmood S, Bilal UA, Ullah RS, Khan A, Nazir A. Advances in chemical modifications of starches and their applications, *Carbohydrate Research*, 476:12–35, 2019.

doi: 10.1016/j.carres.2019.02.007

[7] Li L, Hong Y, Gu Z, Cheng L, Li Z, Li Caiming. Effect of a dual modification by hydroxypropylation and acid hydrolysis on the structure and rheological properties of potato starch, *Food Hydrocolloids*, 77:825–833, 2018.

doi: 10.1016/j.foodhyd.2017.11.026

[8] Kim HY, Jane J lin, Lamsal B. Hydroxypropylation improves film properties of high amylose corn starch, *Industrial Crops and Products*, 95:175–183, 2017.

doi: 10.1016/j.indcrop.2016.10.025

[9] Wang L, Liu X, Wang J. Structural properties of chemically modified Chinese yam starches and their films, *International Journal of Food Properties*, 20(6):1239–1250, 2017.

doi: 10.1080/10942912.2016.1209775

[10] Paramitasari D, Musa M, Putra ON, Suparman S, Pramana YS, Elisa S, Hidayat T, Tjahjono AE, Meidiawati DP, Pudjianto K, Supriyanti A. Hydroxypropylation for functional enhancement of sago starch: The effects of low propylene oxide concentration using response surface methodology, *Journal of Agriculture and Food Research*, 15:100933, 2024.

doi: 10.1016/j.jafr.2023.100933

[11] Calderón-Castro A, Jacobo-Valenzuela N, Félix-Salazar LA, Zazueta-Morales J de J, Martínez-Bustos F, Fitch-Vargas PR, Carrillo-López A, Aguilar-Palazuelos E. Optimization of corn starch

acetylation and succinylation using the extrusion process, *Journal of Food Science and Technology,* 56(8):3940–3950, 2019.

doi: 10.1007/s13197-019-03863-x

[12] Suri S, Singh A. Modification of starch by novel and traditional ways: influence on the structure and functional properties, *Sustainable Food Technology*, 1(3):348–362, 2023.

doi: 10.1039/d2fb00043a

[13] Núñez-Bretón LC, Torres-González CE, Del Ángel-Zumaya JA, Peredo-Lovillo A, Rivera-Villanueva JM, Perea-Flores MJ, Guzmán-Gerónimo RI, Manero O, González- Jiménez FE. Functionalization of starches from Mexican Oxalis tuberosa using dual chemical modification, *Food Hydrocolloids,* 149:109500, 2024.

doi: 10.1016/j.foodhyd.2023.109500

[14] Basilio-Cortés UA, González-Cruz L, Velazquez G, Teniente-Martínez G, Gómez-Aldapa CA, Castro-Rosas J, Bernardino-Nicanor A. Effect of dual modification on the spectroscopic, calorimetric, viscosimetric and morphological characteristics of corn starch, *Polymers,* 11(2):333, 2019.

doi: 10.3390/polym11020333

[15] Zehra N, Ali TM, Hasnain A. Combined effects of hydroxypropylation and alcoholic alkaline treatment on structural, functional and rheological characteristics of sorghum and corn starches, *International Journal of Biological Macromolecules*, 174:22–31, 2021.

doi: 10.1016/j.ijbiomac.2021.01.096

[16] Moin A, Ali TM, Hasnain A. Thermal, morphological, and physicochemical characteristics of succinylated–crosslinked rice starches, *Cereal Chemistry*, 96(5):885–894, 2019.

doi: 10.1002/cche.10191

[17] Mehfooz T, Ali TM, Hasnain A. Effect of cross-linking on characteristics of succinylated and oxidized barley starch, *Journal of Food Measurement and Characterization,* 13(2):1058–1069, 2019.

doi: 10.1007/s11694-018-00021-3

[18] Shaikh M, Ali TM, Hasnain A. Post succinylation effects on morphological, functional and textural characteristics of acid-thinned pearl millet starches, *Journal of Cereal Science,* 63:57–63, 2015.

doi: 10.1016/j.jcs.2015.02.006

[19] Iftikhar SA, Chakraborty S, Dutta H. Effect of acetylation, hydroxypropylation and dual acetylation-hydroxypropylation on physicochemical and digestive properties of rice starches with different amylose content, *Biointerface Research in Applied Chemistry,* 12(5):6788–6803, 2022.

doi: 10.33263/BRIAC125.67886803

[20] Chen P, Zhang Y, Qiao Q, Tao X, Liu P, Xie F. Comparison of the structure and properties of hydroxypropylated acid-hydrolysed maize starches with different amylose/amylopectin contents, *Food Hydrocolloids*, 110:106134, 2021.

doi: 10.1016/j.foodhyd.2020.106134

[21] Javadian N, Mohammadi Nafchi A, Bolandi M. The effects of dual modification on functional, microstructural, and thermal properties of tapioca starch, *Food Science and Nutrition*, 9(10):5467–5476, 2021.

doi: 10.1002/fsn3.2506

[22] Moin A, Ali TM, Hasnain A. Characterization and utilization of hydroxypropylated rice starches for improving textural and storage properties of rice puddings, *International Journal of Biological Macromolecules*, 105:843–851, 2017.

doi: 10.1016/j.ijbiomac.2017.07.109

[23] Ariyantoro AR, Katsuno N, Nishizu T. Effects of dual modification with succinylation and annealing on physicochemical, thermal and morphological properties of corn starch, *Foods*, 7(9):133, 2018.

doi: 10.3390/foods7090133

[24] Jung YS, Lee BH, Yoo SH. Physical structure and absorption properties of tailor-made porous starch granules produced by selected amylolytic enzymes, *PLoS ONE*, 12(7):e0181372, 2017.

doi: 10.1371/journal.pone.0181372

[25] Dhull SB, Bangar SP, Deswal R, Dhandhi P, Kumar M, Trif M, Rusu A. Development and characterization of active native and cross-linked pearl millet starch-based film loaded with fenugreek oil, *Foods*, 10(12):3097, 2021.

doi: 10.3390/foods10123097

[26] Khan A, Saini CS. Effect of roasting on physicochemical and functional properties of flaxseed flour, *Cogent Engineering*, 3(1):1145566, 2016.

doi: 10.1080/23311916.2016.1145566

[27] Zehra N, Ali TM, Hasnain A. Combined effects of hydroxypropylation and alcoholic alkaline treatment on structural, functional and rheological characteristics of sorghum and corn starches, *International Journal of Biological Macromolecules*, 174:22–31, 2021.

doi: 10.1016/j.ijbiomac.2021.01.096

[28] He D, Lv Y, Tong Q. Succinylation improves the thermal stability of egg white proteins, *Molecules*, 24(20):3783, 2019.

doi: 10.3390/molecules24203783

[29] United States Food and Drugs Administration. Food additives permitted for direct addition to food for human consumption, *United States of America: C.F.R*, 21 CFR §172.892, 2023.

[30] Moin A, Ali TM, Hasnain A. Effect of succinylation on functional and morphological properties of starches from broken kernels of Pakistani Basmati and Irri rice cultivars, *Food Chemistry*, 191:52–58, 2016.

doi: 10.1016/j.foodchem.2015.03.119

[31] Mehboob S, Mohsin Ali T, Shaikh M, Hasnain A. Effects of varying levels of succinylation and hydroxypropylation on functional, thermal and textural characteristics of white sorghum starch, *Cereal Chemistry*, 98(3):624–633, 2021.

doi: 10.1002/cche.10406

[32] Lee HL, Yoo B. Effect of hydroxypropylation on physical and rheological properties of sweet potato starch, *LWT*, 44(3):765–770, 2011.

doi: 10.1016/j.lwt.2010.09.012

[33] Marta H, Hasya HNL, Lestari ZI, Cahyana Y, Arifin HR, Nurhasanah S. Study of changes in crystallinity and functional properties of modified sago starch (Metroxylon sp.) using physical and chemical treatment, *Polymers*, 14(22):4845, 2022.

doi: 10.3390/polym14224845

[34] Vaitkeviciene R, Bendoraitiene J, Degutyte R, Svazas M, Zadeike D. Optimization of the sustainable production of resistant starch in rice bran and evaluation of its physicochemical and technological properties, Polymers, 14(17):3662, 2022.

doi: 10.3390/polym14173662

[35] 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*, 80:97–110, 2018.

doi: 10.1016/j.foodhyd.2018.01.032

[36] Sayka RA, Rozada TC, Lima D, Pessôa CA, Viana AG, Fiorin BC. Synthesis and spectroscopic characterization of an unusual succinylated starch applied to carbon paste electrodes, *Starch/Staerke*, 72(3–4), 2020.

doi: 10.1002/star.201900056

[37] Bajer D. Hypophosphite cross-linked starch succinate/chitosan membranes as alternative for packaging and pharmaceutical application, *International Journal of Biological Macromolecules*, 249:126103, 2023.

doi: 10.1016/j.ijbiomac.2023.126103

[38] Zhang W, Zhao G, Huang B, He R, Zhai L, Yang L. Effects of dual modification by cationization and acetylation on the physicochemical and structural characteristics of glutinous rice starch, *International Journal of Biological Macromolecules*, 255:128277, 2024.

doi: 10.1016/j.ijbiomac.2023.128277

[39] Raji AO, Ismael SD, Sani M, Raji MO, Adebayo OF. Value addition and influence of succinylation levels on the quality of wild yam (Discorea villosa) starch, *Food and Function*, 11(9):8014–8027, 2020.

doi: 10.1039/d0fo01482f

Mejora de las características funcionales del almidón de sagú mediante modificación química dual por hidroxipropilación y succinilación

Resumen: El almidón de sagú es un almidón localmente abundante y autóctono de Indonesia. A pesar de su abundancia, está subutilizado y restringido a aplicaciones alimentarias y de empaque debido a sus limitadas características funcionales. El valor del almidón de sagú sin procesar puede incrementarse mediante modificaciones que mejoren su funcionalidad, como la modificación química dual. Este sofisticado proceso es más efectivo que la modificación simple y hace que el almidón sea apto para un rango más amplio de aplicaciones. El objetivo de nuestro estudio fue determinar si una modificación química dual que involucraba hidroxipropilación y succinilación optimizaba las propiedades funcionales del almidón de sagú. Primero, modificamos el almidón de sagú mediante hidroxipropilación con 7 % (p/p) de óxido de propileno en condiciones alcalinas durante 3 horas. Este proceso resultó en almidón hidroxipropilado con un grado de sustitución de 0.107. El segundo paso fue someter el almidón resultante a succinilación utilizando anhídrido succínico a diferentes concentraciones (entre 1 % y 5 % del peso del almidón) en una solución alcalina durante 2 horas. El almidón de sagú modificado dualmente con 3 % (p/p) de anhídrido succínico presentó características funcionales óptimas. El grado de sustitución succinil, la capacidad de retención de agua, la capacidad de retención de aceite, el poder de hinchamiento y la solubilidad del almidón modificado dualmente fueron 0.093, 4.16 $\text{g}\,\text{g}^{-1}$, 7.20 $\text{g}\,\text{g}^{-1}$, 34.25 $\text{g}\,\text{g}^{-1}$ y 16.55 %, respectivamente. Realizamos análisis de propiedades de pegajosidad, espectroscopía infrarroja y análisis de estructura morfológica para determinar los cambios en las características del almidón de sagú después de la hidroxipropilación y la succinilación. La modificación química dual mejoró exitosamente las características funcionales del almidón de sagú, particularmente su capacidad anfifílica y su poder de hinchamiento. Estos resultados respaldan la necesidad de continuar con la investigación y el desarrollo de estos procesos en aplicaciones comerciales.

Palabras Clave: Almidones anfifílicos;Anhídrido succínicoEsterificación;Eterificación;Óxido de propileno

Melhoradas características funcionais do amido de sagu através da modificação química dupla por hidroxi propilação e succinilação

Resumo: O amido de sagu é um amido localmente abundante e autóctone da Indonésia. A pesar de sua abundância, está subutilizado e restrito a aplicações alimentares e de embalagem devido às suas limitadas características funcionais. O valor do amido de sagu sem processar pode se aumentar a través de modificações que melhorem sua funcionalidade, como a modificação química dupla. Este processo sofisticado é mais eficaz do que a modificação simples e torna o amido apto para uma gama mais ampla de aplicações. O objetivo do nosso estudo foi determinar se uma modificação química dupla que envolvia hidroxipropilação e succinilação otimizava as propriedades funcionais do amido de sagu. Primeiro, o amido de sagu foi modificado por hidroxipropilação com 7 % (p/p) de óxido de propileno em condições alcalinas durante 3 horas. Este processo resultou em amido hidroxipropilado com um grau de substituição de 0.107. O segundo passo foi submeter o amido resultante a succinilação utilizando anidrido succínico em diferentes concentrações (entre 1 % e 5 % do peso do amido) em uma solução alcalina durante 2 horas. O amidode sagu modificado duplamente tratado com 3 % (p/p) de anidrido succínico a presentou características funcionais ótimas. O grau de substituição succinil, a capacidade de retenção de água, a capacidade de retenção de óleo, o poder de inchamento e a solubilidade do amido modificado duplamente foram 0.093, 4.16 $\mathrm{g\,g^{-1}}$, 7.20 $\mathrm{g\,g^{-1}}$, 34.25 $\mathrm{g\,g^{-1}}$ e 16.55 %, respectivamente. Realizamos análises das propriedades de gelatinização, espectroscopia infravermelha e análise da estrutura morfológica para determinar as mudanças nas características do amido de sagu após a hidroxipropilação e a succinilação. A modificação química dupla melhorou com sucesso as características funcionais do amido de sagu, particularmente sua capacidade anfifílica e seu poder de inchamento. Esses resultados respaldam a necessidade de continuar a pesquisa e o desenvolvimento desses processos em aplicações comerciais.

Palavras-chave: Almidões anfifílicos; Anidrido succínico; Esterificação; Eterificação; Óxido de propileno

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