

Effects of chemically modified Acorn starch on its functional characteristics and the sustained release properties of monolithic tablets

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Academic Editor: Prof. Constantino Fadda—University of Sassari, Italy

Received: 4 February 2025; Accepted: 1 May 2025; Published: 1 July 2025

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PAPER

Abstract

This study investigated the various chemical modifications that affect the functional properties of Acorn starch. Acorn starch was modified using octenyl succinic anhydride, sodium trimetaphosphate (STMP), oxidation, and lactic acid at different concentrations, with their impacts assessed at various temperatures. Octenyl succinic anhydride-treated samples exhibited swelling power ranging from 2.9% at 60°C to 10.4% at 90°C. Modification levels significantly decreased swelling power ($P < 0.05$), due to structural changes in amylose and amylopectin and stabilization of starch granules from cross-linking. Pasting viscosities increased with modification levels; for example, peak viscosity rose from 1736.0 to 2033.0 cP with octenyl succinic anhydride modification from 3.0 to 9.0%. Lactic acid modification resulted in the highest peak viscosities, whereas STMP cross-linkage showed the lowest. The highest water-holding capacity (WHC) of 139.8% was found with octenyl succinic anhydride at 45°C, while STMP at 0.5% had the lowest WHC. Syneresis varied from 6.80 to 25.30% across freeze–thaw cycles, being lowest with oxidation and highest with STMP. Chemical modifications generally reduced dissolution rates, with lactic acid-modified starch showing the lowest dissolution (58.7%) after 24 h. Results indicated that chemical modifications significantly impact the properties of Acorn starch, emphasizing the need for careful selection based on application.

Keywords: acorn starch; functional properties; monolithic tablets; starch modification

Introduction

It is estimated that more than 15 million oak trees exist within the geographical confines of Jordan, predominantly in the northern and central regions of the nation (Ajo *et al.*, 2020). The mean yield of these trees is assessed to surpass 10 kg per tree, yielding an estimated annual output of 150,000 metric tons, the majority of which remains unutilized for human consumption (Ajo *et al.*, 2020;

Al-Jassim *et al.*, 1998). Nonetheless, the consumption of milled oak flour (i.e., Acorn) for human dietary purposes has experienced a recent uptick, attributable to its substantial nutritional benefits. Saffarzadeh *et al.* (1999), for instance, reported that qualitative and quantitative Acorn amino acid analyses suggest that Acorn protein is more nutritious than that of pecans. Ozcan *et al.* (2023) have specifically reported that Acorn contains significant amounts of minerals, including calcium, phosphorus,

potassium, and niacin. Acorn also contains tannins and flavonoids with additional compounds containing considerable amounts of antioxidants (Ozcan *et al.*, 2023). Moreover, recent investigations connected the antioxidant activity and polyphenols for several herbs used in fighting cancer (Rakic *et al.*, 2006). More specifically, the authors indicated that the ethanolic extracts of thermally treated Acorn have increased protective properties in lipid deposition, help control obesity, and aid in protecting from diabetic complications, therefore, suggesting that modifications would change the functional characteristics of Acorn starch.

Starch is extensively used in the food industry to provide diverse functionalities, such as gelling and thickening agents. Starch in its native form usually cannot withstand the severe processing and storage conditions, therefore modifications such as cross-linking and substitution are often applied to improve existing and/or impart new functionalities (Mhaske *et al.*, 2023; Wang *et al.*, 2022). Chemically modified starches play a crucial role in enhancing the functional characteristics of starches for various applications, particularly in the food industry (Garcia *et al.*, 2020; Konował *et al.*, 2024). Different modification techniques, such as oxidation, esterification, and cross-linking, significantly alter the physicochemical properties of starch, leading to improved functionalities (Konował *et al.*, 2024; Wang *et al.*, 2022). For instance, ultrasonic and chemical dual modifications enhanced swelling power and solubility while improving freeze-thaw stability, making starch more versatile for food applications (Ashogbon 2021). The authors reported that doubly modified waxy corn starch exhibited a WBC of 206.3%, surpassing native starch. Similarly, Hadi *et al.* (2024) indicated that short-chain fatty acid modification reduces gelatinization temperatures and increases resistant starch content, which is beneficial for health-oriented food products. Others, however, indicated that cross-linking restricted swelling power, which is essential for applications such as food packaging (Iqbal *et al.*, 2024). Generally, starch is modified chemically, physically, and enzymatically and/or genetically (Bangar *et al.*, 2022; Rakic *et al.*, 2006).

Although considered as harsh treatments, starch chemical modifications are considered to be more effective than physical and/or enzymatic methods in structural alterations and introduction of new functional groups (Bangar *et al.*, 2022; Bharati, 2023). Chemical modifications have effects on the physicochemical properties of starch and make it fit for various industrial uses with their significantly altered physicochemical properties (Bisht, 2018). The modification of native granular starches alters their behavior in terms of gelatinization, pasting, and retrogradation (Subroto *et al.*, 2021). Subroto *et al.* (2021) reported improved rheological properties of

cross-linked sweet potato starch as a fat replacer to produce cream, ice cream, mayonnaise, muffins, and other products. Meifang *et al.* (2019) also reported that sweet potato starch subjected to cross-linking resulted in resistant starch production. Heo *et al.* (2017) reported that cross-linking with sodium trimetaphosphate (STMP) or sodium tripolyphosphate (STPP) improved the rheological and pasting properties of potato starch.

There are limited studies on the effects of modifying Acorn starches on their functional characteristics, especially in pharmaceutical applications. Therefore, this study aims to investigate the effects of various modifications of Acorn starches on their functional characteristics.

Materials and Methods

Acorn and Acorn starch extraction

Acorn starch was extracted from oak (*Quercus calliparinos*) collected by hand from the northern region of Jordan. The process began immediately after harvesting, with the oak being washed with tap water, soaked for 24 h, and then washed again. The oak was sun-dried for 72 h, peeled, and the Acorn flour was ground and sifted through a US 100-mesh screen (149 μm).

Starch extraction was performed using a sodium hydroxide solution, following the method of Quan *et al.* (1997), with some modifications to the soaking times. First, the Acorn flour was defatted by soaking it in hexane (1:10 w/v) for 24 h, after which the hexane was drained. The defatted flour was then soaked in 0.2 N sodium hydroxide solution (1:10 w/v) for 2 h at 25°C with continuous stirring. The mixture was subsequently wet-milled in an Osterizer blender for 3 min at speed setting 6 and filtered twice through US standard test sieves number 100 and 400. The resulting slurry was centrifuged at 4000 rpm for 30 min at 4°C using a 5810R centrifuge (Eppendorf, Germany), and the supernatant was discarded. The sediment was washed five times with 0.2 N sodium hydroxide (1:10 flour to sodium hydroxide ratio) and centrifuged again under the same conditions. The dark tailings on top of the starch sediment were carefully removed and discarded. This process was repeated with three washes using distilled water, followed by centrifugation and removal of the remaining dark tailings. Finally, the washed sediment was suspended in distilled water and its pH was adjusted to 7.0 using 0.1 N hydrochloric acid (HCl), as measured with a pH meter (model 40675/0001: HANNA Instruments, UK). A final centrifugation at 4000 rpm for 30 min was performed, and the starch was then air-dried at 40°C for 48 h to achieve a moisture content of 12% before being ground and sifted through a US standard sieve number 100.

Acorn starch modifications

Sodium trimetaphosphate cross-linkage modification

The cross-linked Acorn starch was prepared by reacting native Acorn starch samples with POCl₃ according to the procedure described by Zheng *et al.* (1999). Native starch samples (450 g) were dispersed in 780 mL distilled water containing 20% Na₂SO₄ and stirred to obtain a uniform suspension. The pH of the suspension was adjusted to 10.5 with 4% NaOH. POCl₃ was added at 0.01, 0.1, and 0.5% concentrations, based on the dry weight of Acorn starch. The mixture was left to react for 1 h with stirring. The pH of the mixture was adjusted to 5.5 with 15% HCl, and after sedimentation, the modified starch was washed thrice with distilled water before drying at 40°C. The dried cross-linked starch samples were ground and passed through a 100-mesh standard sieve.

Octenyl succinic anhydride (OSA) modification

Acorn starch succinylation was performed according to Lawal *et al.* (2015) with modifications of the level of octenyl succinic anhydride. In summary, Acorn starch samples (50 g, dry basis) and distilled water (140 mL) were added into a round-bottom flask and mixed with a magnetic stirrer to adjust the pH to 8.5 using 1 N NaOH. Octenyl succinic anhydride (1, 3, and 5% based on the weight of Acorn starch) was added (0.1 mL/min) with continuous stirring. The mixture was then refluxed for 6 h at 115°C and cooled to room temperature (22.2°C). After modification, the Acorn starch reactant was adjusted to pH 7, washed, and centrifuged (3000 g for 10 min). Thereafter, the OSA starch was dried at 40°C before passing through a 100-mesh sieve.

Oxidation

Acorn starch oxidation was performed according to the method described by Dias *et al.* (2011). Acorn starch sample (200 g, dry basis) was suspended in 500 mL of distilled water in a 1 L glass reactor, heated at 40°C, and subjected to sodium hypochlorite treatment (i.e., active chlorine concentrations of 0.5, 1.0, and 1.5 g/100 g). The pH of the starch slurry was adjusted to 7.0. After the addition of sodium hypochlorite, the pH value of the slurry was maintained at 7.0 for 60 min. The slurry was then filtered and washed with a twofold volume of distilled water and then dried at 40°C. The dried Acorn starch samples were then ground and passed through a 100-mesh standard sieve.

Acid modification

The acid modification of Acorn starch was carried out according to Tavares *et al.* (2010). A sample of starch (300 g, dry basis) was dispersed in 900 mL of water in a glass reactor maintained at 40°C with constant stirring. Lactic acid was added with concentrations of 3, 5, or 10 g/100 g starch (d.b), and the mixture was stirred for 1 h.

The dispersion was then neutralized with sodium hydroxide (1 mol/L), filtered, and washed with distilled water (1:2). Starch was then dried at 40°C before grinding and passing through a 100-mesh standard sieve.

Functional characteristics of Acorn starch

Water holding capacity

Water holding capacity (WHC; %) of each treatment was determined by Saleh *et al.* (2016). In summary, duplicate flour treatments were dispersed in distilled water, and the dispersions were allowed to stand for 1 h at 25, 35, and 45°C before centrifuging at 3800 rpm for 30 min. Sediment weights were recorded and used to calculate WHC (%) using the following equation.

$$\text{WHC (\%)} = \frac{\text{Weight of Sediment}}{\text{Weight of Dry Solids}} \times 100\%$$

Pasting measurement

Pasting viscosities, including peak, trough, setback, breakdown, and final viscosities, as well as pasting temperature, were measured using a Rapid Visco Analyzer according to AACCI method 76-21 (AACCI International, 2010). In brief, approximately 3 g of each sample (adjusted for moisture content) was mixed with 25 mL of distilled water. The resulting slurry was heated to 50°C and mixed at 160 rpm for 1 min before being gradually heated from 50 to 95°C at a rate of 12°C/min. The paste was then maintained at 95°C for 2.5 min and cooled back to 50°C at the same rate of 12°C/min. Data from the RVA were analyzed using Thermocline version 1.2 software, and pasting properties were measured in duplicate.

Freeze–thaw stability (syneresis)

To assess the freeze–thaw stability, aqueous dispersions of the treatments (5 g treatment per 100 g distilled water) were prepared and gelatinized at 95°C with continuous shaking for 30 min. After gelatinization, the samples were cooled to 25°C and subjected to freeze–thaw cycles. The gelatinized treatments were frozen at –20°C for 24 h, thawed at 30°C for 2 h, and then centrifuged at 3800 rpm for 30 min. The supernatants from each cycle were weighed to determine the degree of syneresis, which was expressed as a percentage of freeze stability. Three measurements per cycle were taken, and results were averaged for each cycle (Saleh *et al.*, 2018).

Swelling power

Forty milligrams of starch was placed in a 2 mL micro-centrifuge tube, and 1.5 mL of deionized water was added. The starch was allowed to swell in a heat block set at 60, 70, 80, and 90°C for 1 h, and was then rapidly cooled in an ice bath. The tube was centrifuged in an

Eppendorf Centrifuge 5415D (Germany) at $12,000 \times g$ for 10 min. After centrifugation, excess water was carefully removed, and the remaining gel was weighed. The swelling power was calculated as follows:

$$\text{Swelling Power (g/g)} = \frac{\text{Gel Weight (g)}}{\text{Dry Starch Weight (g)}}$$

Monolithic tablets production and properties

Preparation of tablets

Starch and propranolol hydrochloride (30% loading w/w of tablet) were mixed in a mini-manual mixer (Inversina, Bioengineering AG, Wald, Switzerland) for 10 min. Magnesium stearate (1% w/w of tablet) was added as a lubricant, and the mixture was mixed for another minute. Tablets were prepared by compressing 500 mg of the mixture at 2.0 MT with a 13mm die using a hydraulic press (Carver, Wabash, IN).

Drug release was studied using Apparatus II dissolution equipment (Varian, Cary, NC). Tablets were immersed in 900 mL of deionized water at 37.5°C for 24 h at a paddle rotation speed of 50 rpm. Five milliliter samples of the dissolution medium were taken without medium replacement, and drug release was measured using a spectrophotometer (Beckman Coulter, Fullerton, CA) at 290 nm. All experiments were performed in triplicate.

Statistical analysis

Data were analyzed using JMP version 10.0 (SAS Institute, Cary, NC, 2010). The analysis of variance (ANOVA) of the different treatments was done using Student's *t*-test, in which the rheological properties and chemical characteristics were used as the sources of variation. The means of two replicates from each treatment variable were compared for significance at a 5% probability using the Least Significant Difference test (LSD).

Results and Discussion

Swelling power of modified Acorn starch

Our swelling power results were significantly greater than these values, indicating the crosslinking modifications of sweet potato starch (i.e., 3.40–3.67 g/g) (Marta and Tensiska, 2017). This observation implies that the cross-linking modifications utilized in our investigation may have induced more profound structural alterations within the starch granules. Specifically, the reorganization of amylose and amylopectin chains stemming from

these modifications could have intensified the cohesion among starch granules, ultimately resulting in the increased swelling power we observed (Chen *et al.*, 2017). These structural reconfigurations, especially the reorientation of polymer chains, augment the interactions between starch molecules, thereby facilitating enhanced water retention and granule expansion during the gelatinization process. Moreover, the augmented stability of the starch granules may be ascribed to the improved liberation of double helix bonds within the crystalline domains of the granules, in contrast to their unmodified counterparts. Marta and Tensiska (2017) assert that these bonds are pivotal in preserving the structural integrity of the starch granules, and their regulated release during cross-linking may contribute to an enhanced swelling capacity. In this context, cross-linking fortifies the hydrogen bonds that maintain the cohesion of the granules, culminating in superior granule stability and enhanced swelling during the gelatinization phase. The magnitude of this effect is intricately linked to the degree of cross-linking, as moderate cross-linking reinforces the granules without undermining their capacity to absorb water (Kim and Yoo, 2010).

The augmentation of swelling power observed in cross-linked starches can also be attributed to the development of a rigid outer crust on the starch granules, which facilitates granule enlargement (Hazarika and Sit, 2016). This external layer serves as a protective barrier, inhibiting premature disintegration of the granules while concurrently permitting water infiltration to instigate swelling. The structural soundness of this crust is essential for regulated swelling, as it promotes gradual water absorption and assists in maintaining the granule morphology during the gelatinization process. Nonetheless, an increase in the modification level may lead to a decline in swelling power due to excessive cross-linking. This decline can be elucidated by the over stabilization of the granule structure resulting from the excessive liberation of double helix bonds and the reinforcement of hydrogen bonds, which culminates in a more inflexible starch matrix. Excessive cross-linking may impede water penetration, thereby constraining the granule's capacity to swell and expand adequately. This phenomenon has been documented in various studies, wherein an overabundance of cross-linking results in diminished swelling properties as the granule becomes overly compact, precluding sufficient water absorption and expansion. Moreover, the balance between cross-linking and swelling power is critical not only for functional applications but also in determining the end-use properties of modified starches. For instance, studies have shown that while moderate levels of modification can enhance certain characteristics such as thermal stability and water solubility, excessive cross-linking leads to a compromise on textural attributes essential for food products.

This trade-off necessitates careful optimization of processing conditions, such as autoclaving time and the number of cycles, which significantly influence both the swelling capacity and the overall performance of the starch. As researchers continue to explore these parameters, it becomes increasingly clear that achieving an ideal modification level requires a nuanced understanding of the interplay between molecular structure and macroscopic behavior, thereby guiding future innovations in starch-based materials.

Table 1 shows a comparison between various modifications as well as the untreated Acorn starch as a control. Our results are in agreement with Carmona-Garcia *et al.* (2009), who reported an increase in swelling power with temperature. Oxidation treatment resulted in the highest swelling power at 60°C with swelling power values of 5.0, 3.8, and 4.0% of low (i.e., 0.5), medium (i.e., 1.0), and high (i.e., 1.5) modification levels, respectively. The effect of swelling temperature on the swelling power resulted in diminishing the swelling power trends. For instance, at 80 and 90°C, no significant differences were reported for most modification treatments when swelling power reached values greater than 7%.

Pasting viscosities of modified Acorn starch

Table 2 shows the pasting viscosities of modified Acorn starch. Results showed, irrespective to the modification

level, an increase ($P < 0.05$) in pasting viscosities of modified Acorn starch with the increase in the level of modification. For example, peak viscosity increased from 1736.0 to 2033.0 cP with the increase in octenyl succinic anhydride modification from 3.0 to 9.0%. Similarly, peak viscosity increased from 2839.5 to 3140.0 cP with the increase in lactic acid modification level from 3.0 to 10%. Moreover, final viscosity of Oxidation modified Acorn starch increased ($P < 0.05$) from 3054.0 to 3860.5 cP with the increase in modification level from 0.5 to 1.5%. Similar trends were noticed for trough, setback, and breakdown viscosities.

Results indicated that cross-linking impacted pasting viscosities depending on factors including the starch source (Sudheesh *et al.*, 2020). For instance, the introduction of chemical bonding resulting from the modifications appeared to stabilize starch granules against shear force, and acid and heat treatment lowered their breakdown. For instance, octenyl succinic anhydride and STMP cross-linkage modifications of Acorn starch increased peak viscosities, while oxidation and lactic acid modifications resulted in reduced paste viscosities of Acorn starch compared to the native starch. It is believed that during modifications, the kinetics of amylose leaching, granular swelling, and friction between swollen granules impact the increase in viscosity throughout the heating cycle. In addition, pasting properties are also affected by the rigidity of starch granules, which affects the granule swelling potential (Waterschoot *et al.*, 2016). Additionally, the

Table 1. Swelling power (%) at 60, 70, 80, and 90°C of modified Acorn starch.

Treatment	Level		Swelling power (%) at various temperatures (°C)			
			60	70	80	90
Octenyl succinic anhydride	Low	3	3.2 ± 0.02 ^{Dc}	5.7 ± 0.11 ^{Cb}	8.7 ± 0.17 ^{Ba}	10.4 ± 0.37 ^{Aa}
Sodium trimetaphosphate (STMP) cross-linkage		0.05	3.7 ± 0.03 ^{Db}	4.9 ± 0.06 ^{Cc}	8.7 ± 0.11 ^{Aa}	9.6 ± 0.01 ^{Ab}
Oxidation		0.5	5.0 ± 0.05 ^{Ba}	5.2 ± 0.07 ^{Cb}	8.7 ± 0.73 ^{Aa}	10.0 ± 0.42 ^{Aa}
Lactic acid	Medium	3	3.5 ± 0.01 ^{Db}	6.3 ± 0.09 ^{Ca}	9.9 ± 0.08 ^{Aa}	10.3 ± 0.17 ^{Aa}
Control		–	3.5 ± 0.01 ^{Db}	4.2 ± 0.13 ^{Cd}	6.4 ± 0.33 ^{Bb}	7.1 ± 0.10 ^{Ac}
Octenyl succinic anhydride		6	3.1 ± 0.04 ^{Dd}	5.3 ± 0.05 ^{Cb}	7.5 ± 0.17 ^{Bb}	9.0 ± 0.08 ^{Ac}
STMP cross-linkage	High	0.1	3.2 ± 0.02 ^{Cc}	4.5 ± 0.01 ^{Bc}	8.5 ± 0.01 ^{Ba}	8.8 ± 0.18 ^{Ab}
Oxidation		1	3.8 ± 0.01 ^{Da}	5.3 ± 0.07 ^{Cb}	8.9 ± 0.10 ^{Bb}	10.0 ± 0.25 ^{Aa}
Lactic acid		5	3.2 ± 0.04 ^{Dc}	6.1 ± 0.01 ^{Ca}	8.4 ± 0.07 ^{Ba}	9.4 ± 0.03 ^{Ab}
Control	–	–	3.5 ± 0.02 ^{Db}	4.2 ± 0.13 ^{Cd}	6.4 ± 0.33 ^{Bc}	7.1 ± 0.10 ^{Ad}
Octenyl succinic anhydride	High	9	2.9 ± 0.04 ^{De}	4.7 ± 0.06 ^{Cc}	7.6 ± 0.06 ^{Bc}	9.1 ± 0.32 ^{Aa}
STMP cross-linkage		0.5	3.1 ± 0.03 ^{Dd}	4.0 ± 0.07 ^{Cd}	8.0 ± 0.02 ^{Bc}	8.6 ± 0.01 ^{Ab}
Oxidation		1.5	4.0 ± 0.04 ^{Da}	4.7 ± 0.05 ^{Bc}	7.1 ± 0.15 ^{Bb}	8.8 ± 0.40 ^{Ab}
Lactic acid	High	10	3.0 ± 0.04 ^{Cd}	5.7 ± 0.07 ^{Ba}	8.7 ± 0.05 ^{Ba}	9.2 ± 0.53 ^{Aa}
Control		–	–	3.5 ± 0.02 ^{Dc}	4.2 ± 0.13 ^{Cc}	6.4 ± 0.033 ^{Bd}

¹For treatments at the same set of levels (e.g., low, medium, and high), means of swelling power values with different letter(s) are significantly ($P < 0.05$) different according to least squares differences. STMP, sodium trimetaphosphate.

Table 2. Pasting viscosities (cP) of modified Acorn starch at a set of levels.

Treatment	Level		Pasting viscosities (cP)				
			Peak	Trough	Final	Setback	Breakdown
Octenyl succinic anhydride	Low	3	1736.0 ± 1.4 ^c	1303.0 ± 15.6 ^d	2509.5 ± 9.2 ^c	433.0 ± 17.0 ^b	1206.5 ± 6.4 ^c
Sodium trimetaphosphate (STMP) cross-linkage		0.05	1442.5 ± 31.1 ^e	1287.0 ± 7.8 ^e	2410.5 ± 71.4 ^d	155.5 ± 38.9 ^e	1123.5 ± 79.2 ^d
Oxidation		0.5	2280.5 ± 95.5 ^b	1879.5 ± 70.0 ^a	3054.0 ± 17.8 ^b	401.0 ± 25.5 ^{b,c}	1778.5 ± 66.8 ^a
Lactic acid		3	2839.5 ± 98.3 ^a	1869.5 ± 17.7 ^a	3677.0 ± 11.7 ^a	970.0 ± 80.6 ^a	1807.5 ± 44.0 ^a
Control		--	1865.0 ± 21.2 ^c	1549.0 ± 7.1 ^c	1763.0 ± 4.2 ^d	316.0 ± 28.3 ^c	214.0 ± 2.8 ^d
Octenyl succinic anhydride	Medium	6	2012.5 ± 16.3 ^c	1435.5 ± 30.4 ^d	2745.0 ± 29.7 ^c	577.0 ± 14.1 ^b	1309.5 ± 0.7 ^c
STMP cross-linkage		0.1	1779.0 ± 3.5 ^d	1446.5 ± 18.4 ^d	2718.0 ± 2.1 ^c	332.5 ± 14.8 ^d	1271.5 ± 16.3 ^c
Oxidation		1	2471.5 ± 2.1 ^b	1998.5 ± 9.2 ^a	3658.0 ± 24.7 ^a	473.0 ± 7.1 ^c	1862.0 ± 15.6 ^a
Lactic acid		5	2976.0 ± 80.6 ^a	1854.0 ± 62.2 ^b	3635.0 ± 11.7 ^b	1122.0 ± 18.4 ^a	1781.0 ± 55.2 ^b
Control		--	1865.0 ± 21.2 ^d	1549.0 ± 7.1 ^c	1763.0 ± 4.2 ^e	316.0 ± 28.3 ^d	214.0 ± 2.8 ^e
Octenyl succinic anhydride	High	9	2033.0 ± 32.5 ^c	1489.5 ± 67.2 ^{c,d}	2979.5 ± 9.2 ^b	543.5 ± 34.6 ^c	1490.0 ± 58.0 ^b
STMP cross-linkage		0.5	2255.0 ± 4.2 ^b	1744.5 ± 0.7 ^b	3195.5 ± 4.2 ^b	510.5 ± 3.5 ^b	1451.0 ± 3.5 ^b
Oxidation		1.5	2732.0 ± 80.6 ^b	1715.5 ± 29.0 ^b	3860.5 ± 90.5 ^a	1016.5 ± 51.6 ^a	1338.5 ± 61.5 ^c
Lactic acid		10	3140.0 ± 62.3 ^a	2334.5 ± 26.2 ^a	4939.0 ± 46.7 ^a	805.5 ± 88.4 ^b	2604.5 ± 77.8 ^a
Control		--	1865.0 ± 21.2 ^d	1549.0 ± 7.1 ^c	1763.0 ± 4.2 ^d	316.0 ± 28.3 ^d	214.0 ± 2.8 ^d

¹For treatments at the same set of levels (e.g., low, medium, and high), means of pasting viscosities with different letter(s) are significantly ($P < 0.05$) different according to least squares differences. STMP, sodium trimetaphosphate.

higher peak viscosity values corresponded to a higher thickening power of starch (Chibuzo, 2012). These findings were further evident in the breakdown viscosity results, which is a measure of the fragility of starch (Chen *et al.*, 2019), that was remarkable in octenyl succinic anhydride and STMP cross-linkage modifications compared to oxidation and lactic acid modifications of similar levels (Table 1). The lower the breakdown in pasted starch, the stronger the ability of modified starch to resist physical factors and remain stable due to the reinforcement by the new bonding that restricts the breakdown (Fonseca *et al.*, 2021). The decrease in pasting viscosities of oxidation and lactic acid modifications are in agreement with Vanier *et al.* (2012), who found that the oxidized bean has a lower viscosity than the native starch bean; the partial breaking of glycosidic connections has been attributed to the reduction in peak and final viscosities of oxidized starches. The authors suggested the improvement in amylose chains' structural distance, thus preventing molecular approximation and minimizing retrogradation.

Lactic acid modification resulted in greater peak viscosities compared to octenyl succinic anhydride, STMP cross-linkage, oxidation, and control treatment, irrespective of the level used. For example, at low modification levels, lactic acid treatment had a peak viscosity value of 2839.5 compared with 1736.0, 1442.5, 2280.5, and 1865.0 for octenyl succinic anhydride, STMP cross-linkage, oxidation,

and the control, respectively. Sodium trimetaphosphate cross-linkage modification had the lowest viscosities of 1442.5 and 1779.0 cP when modified at 0.05 and 0.1 levels, respectively. Similar trends are reported for final, setback, and breakdown viscosities. Final viscosity of lactic acid had values of 3677.0, 3635.0, and 4939.0 cP when Acorn starch was modified at 3, 5, and 10% levels, respectively, compared to 1763.0 cP of the nonmodified samples.

Water holding capacity (WHC) of modified Acorn starch

Water holding capacity (%) and syneresis of Acorn starch modified are shown in Table 3. Results showed an increase in WHC ($P < 0.05$) with the increase in holding temperature, with octenyl succinic anhydride level of 3.0% when held at 45°C having the greatest ($P < 0.05$) WHC value of 139.8% compared to the control or nonmodified samples that have a WHC of 127.8%. WHC of octenyl succinic anhydride treatment increased from 127.3 to 139.8% with the increase in holding temperature from 25 to 45°C. Comparing treatments, results indicated that control nonmodified treatments performed at 35 and 45°C had the greatest ($P < 0.05$) WHC of 120.5 and 127.8%, respectively, except for the octenyl succinic anhydride treatment at level 3.0. Additionally, STMP cross-linkage modification at a 0.5% level had the lowest ($P < 0.05$) WHC of 93.0. However, lactic acid modification treatments had the overall lowest WHC.

Table 3. Water holding capacity (WHC, %) at 25, 35, and 45°C of modified Acorn starch at various sets of levels (low, medium, and high).

Treatment	Level		WHC (%) at various temperatures (°C)		
			25	35	45
Octenyl succinic anhydride	Low	3	127.3 ± 3.6 ^{A,a}	135.1 ± 0.7 ^{A,a}	139.8 ± 8.4 ^{A,a}
Sodium trimetaphosphate (STMP) cross-linkage		0.05	125.4 ± 1.9 ^{A,a}	109.3 ± 2.4 ^{B,c,d}	109.7 ± 2.5 ^{B,d}
Oxidation		0.5	113.9 ± 0.8 ^{Ab,c}	116.7 ± 2.0 ^{A,b,c}	115.3 ± 0.2 ^{A,c}
Lactic acid		3	108.8 ± 1.4 ^{A,c}	100.9 ± 0.3 ^{B,d}	110.4 ± 2.5 ^{A,d}
Control		–	117.4 ± 1.2 ^b	120.5 ± 1.8 ^b	127.8 ± 2.4 ^b
Octenyl succinic anhydride	Medium	6	113.9 ± 3.4 ^{B,c,d}	116.7 ± 3.3 ^{A,a}	129.1 ± 1.4 ^{A,a}
STMP cross-linkage		0.1	124.6 ± 3.2 ^{A,a,b}	110.3 ± 2.0 ^{B,b}	99.4 ± 1.8 ^{C,c}
Oxidation		1	129.9 ± 4.4 ^{A,a}	120.1 ± 2.7 ^{B,a}	120.1 ± 1.0 ^{A,b}
Lactic acid		5	108.3 ± 0.3 ^{B,d}	110.4 ± 3.0 ^{B,b}	117.7 ± 1.8 ^{A,b}
Control		–	117.4 ± 1.2 ^{b,c}	120.5 ± 1.8 ^a	127.8 ± 2.4 ^a
Octenyl succinic anhydride	High	9	119.4 ± 2.7 ^{A,a}	122.4 ± 1.4 ^{A,a}	126.2 ± 2.7 ^{A,a,b}
STMP cross-linkage		0.5	122.8 ± 3.4 ^{A,a}	111.5 ± 2.6 ^{B,b}	93.0 ± 2.9 ^{C,c}
Oxidation		1.5	122.6 ± 2.0 ^{A,a}	117.0 ± 0.6 ^{B,a,b}	122.5 ± 0.5 ^{A,b}
Lactic acid		10	111.0 ± 0.3 ^{C,b}	118.4 ± 1.7 ^{B,a}	125.2 ± 1.4 ^{A,a,b}
Control		–	117.4 ± 1.2 ^a	120.5 ± 1.8 ^a	127.8 ± 2.4 ^a

¹For treatments at the same set of levels (e.g., low, medium, and high), means of water holding capacity at various temperatures with different letter(s) are significantly ($P < 0.05$) different according to least squares differences. STMP, Sodium trimetaphosphate.

The variation in modification effects on WHC (%) was attributed to the kinetics of incorporating hydrophilic hydroxypropyl groups in modified starches. The increase in cross-linking level, for example, resulted in the addition of intra- and intermolecular bonds at various positions in starch granules. The minimum value observed was probably due to the excess concentration of the cross-linking agents causing the formation of bulky molecules, which coagulated and limited the cross-linking reaction in starch granules (Yusnita *et al.*, 2019). Mirmoghtadaie *et al.* (2009) reported that the effect of cross-linking could depend on the starch's botanical source and the cross-linking agent used.

More specifically, the decrease in WHC compared to the control (i.e., nonmodified Acorn starch) was attributed to the difficulties of the crystalline lamellae of starch granules to be oxidized (Vanier *et al.*, 2017). Oxidation can affect swelling forces by the increase in the crystalline region and decrease in the amorphous region, thereby reducing the number of available binding sites and lowering the ability of starches to uptake more moisture, and thus decreasing the WHC (Sudheesh *et al.*, 2020).

Freeze–thaw stability of modified Acorn starch

Syneresis ranged from 6.80 to 24.88%, 9.35 to 25.30%, and 9.65 to 25.30% for cycles 1, 2, and 3, respectively (Table 4). Oxidation treatment resulted in significantly ($P < 0.05$) the lowest (i.e., 6.80%) and STMP cross-linkage

treatment resulted in significantly ($P < 0.05$) the greatest (i.e., 25.30%) syneresis value compared to other treatments, regardless of the modification level used.

Syneresis is an essential parameter in gel system stability; it refers to the phenomenon in which the starch gel of the bulk phase water is released from the polymer network structure due to the restructuring of the leached starch molecules after continuous freezing and thawing to form a spongy texture (Ding *et al.*, 2020). Majzoobi *et al.* (2016) studied the effect of acetic acid on the physical properties of pregelatinized wheat and corn starch gels using different concentrations (0.01, 0.05, 1, and 10%) and reported an overall syneresis range from 17.23 to 32.67%, similar to the results reported in this study. The authors related the amount of water released to increased intermolecular and intramolecular hydrogen bonding due to the interaction between starch chains (amylose–amylose, amylose–amylopectin, and amylopectin–amylopectin) during frozen storage. In the same manner, the acetyl groups present during acetylation facilitate the prevention of the realignment and association between macromolecules, thereby leading to reduced syneresis (Singh *et al.*, 2012). The more released water in acetylation modifications compared to the control can also be related to the amount of water absorbed and the formation of smaller starch molecules of lactic acid-treated Acorn starch.

The increased stability of cross-linked starch during freeze–thaw cycles was attributed to the introduction of phosphate groups (Zhao *et al.*, 2015). The phosphate

Table 4. Syneresis (i.e., Cycles 1, 2, and 3) for modified Acorn starch at various sets of levels (low, medium, and high).

Treatment	Level		Syneresis (%)		
			Cycle 1	Cycle 2	Cycle 3
Octenyl succinic anhydride	Low	3	9.55 ± 0.57 ^d	13.55 ± 0.49 ^c	13.55 ± 0.49 ^c
Sodium trimetaphosphate (STMP) cross-linkage		0.05	24.55 ± 0.01 ^a	25.30 ± 0.42 ^a	25.30 ± 0.57 ^a
Oxidation		0.5	9.50 ± 0.21 ^d	13.50 ± 0.21 ^c	13.50 ± 0.21 ^c
Lactic acid		3	16.50 ± 0.21 ^b	20.50 ± 0.42 ^b	21.60 ± 0.42 ^b
Control		–	12.75 ± 0.35 ^c	14.50 ± 0.01 ^c	15.10 ± 0.14 ^c
Octenyl succinic anhydride	Medium	6	12.30 ± 0.49 ^c	15.85 ± 0.64 ^b	15.95 ± 0.64 ^b
STMP cross-linkage		0.1	20.20 ± 0.64 ^a	21.45 ± 1.27 ^a	21.75 ± 1.27 ^a
Oxidation		1	6.80 ± 0.57 ^d	9.35 ± 0.14 ^c	9.65 ± 0.14 ^c
Lactic acid		5	15.35 ± 2.12 ^b	21.75 ± 0.71 ^a	22.80 ± 0.57 ^a
Control		–	12.75 ± 0.35 ^c	14.50 ± 0.01 ^b	15.10 ± 0.14 ^b
Octenyl succinic anhydride	High	9	9.30 ± 0.28 ^d	13.35 ± 0.49 ^d	13.35 ± 0.64 ^d
STMP cross-linkage		0.5	22.80 ± 0.14 ^a	25.00 ± 0.49 ^a	25.10 ± 0.49 ^a
Oxidation		1.5	8.55 ± 0.57 ^d	10.65 ± 0.92 ^e	10.65 ± 0.78 ^e
Lactic acid		10	14.95 ± 0.49 ^b	20.40 ± 0.35 ^b	21.90 ± 0.28 ^b
Control		–	12.75 ± 0.35 ^c	14.50 ± 0.01 ^c	15.10 ± 0.14 ^c

¹For treatments at the same set of levels (e.g., low, medium, and high), means of syneresis with different letter(s) are significantly ($P < 0.05$) different according to least squares differences.
STMP, Sodium trimetaphosphate.

groups were positively charged and repelled each other. As a result, starch molecules swelled and the size of starch granules increased (Liu *et al.*, 2014). Furthermore, cross-linked starch tends to reduce the proportion of syneresis in starch, preventing it from retrograding. In addition, the cross-linked starch will prevent the amylose chain from reassociating by blocking the starch chain with the newly added functional group.

The variation in syneresis was attributed to the variation in starch type as well as the amount of amylose and amylopectin in each type of starch used (Shang, 2021). More specifically, oxidized starch with a low syneresis value indicates slow retrogradation of starch gels due to strong interactions between dispersed amylose or amylopectin and water molecules (Carmona-Garcia *et al.*, 2009). Matsuguma *et al.* (2009), on the other hand, reported that oxidized starch has a high value rather than native starch and suggested that the increase in syneresis intensity could be associated with the amylose content, degree of starch polymers association, length of amylopectin side chains, and the degree of polymerization of amylose and amylopectin.

Sustained release of Monolithic tablets

Figure 1 shows the per cent dissolution over 24 h for monolithic tablets prepared using both modified and

nonmodified Acorn starch. Results revealed that Acorn chemical modifications significantly ($P < 0.05$) influenced the release kinetics, generally leading to a reduction in dissolution rate compared to the native starch. Specifically, nonmodified Acorn starch exhibited the highest dissolution (i.e., 99.0%) after 24 h, followed by tablets containing STMP cross-linked (i.e., 99.0%), octenyl succinic anhydride-treated (i.e., 97.0%), oxidized (i.e., 86.9%), and lactic acid-modified starch (i.e., 58.7%).

These findings highlight notable differences in the release-extending properties imparted by each type of starch modification. The reduced dissolution observed with lactic acid treatment suggests a stronger matrix formation, which may be advantageous for applications requiring slower drug release. Such variation underscores the impact of starch modification on the excipient's functionality in sustained release formulations.

The differences in release behavior can be attributed to the physicochemical alterations introduced by each modification, particularly in terms of swelling capacity and pasting properties (Fonseca *et al.*, 2021). For instance, cross-linking reactions appeared more prevalent within the amorphous regions of native starch granules, leading to uneven distribution of cross-linked zones. This heterogeneity likely disrupted the integrity of the tablet matrix, thereby affecting its release performance. In contrast, cross-linking in the gelatinized state facilitated

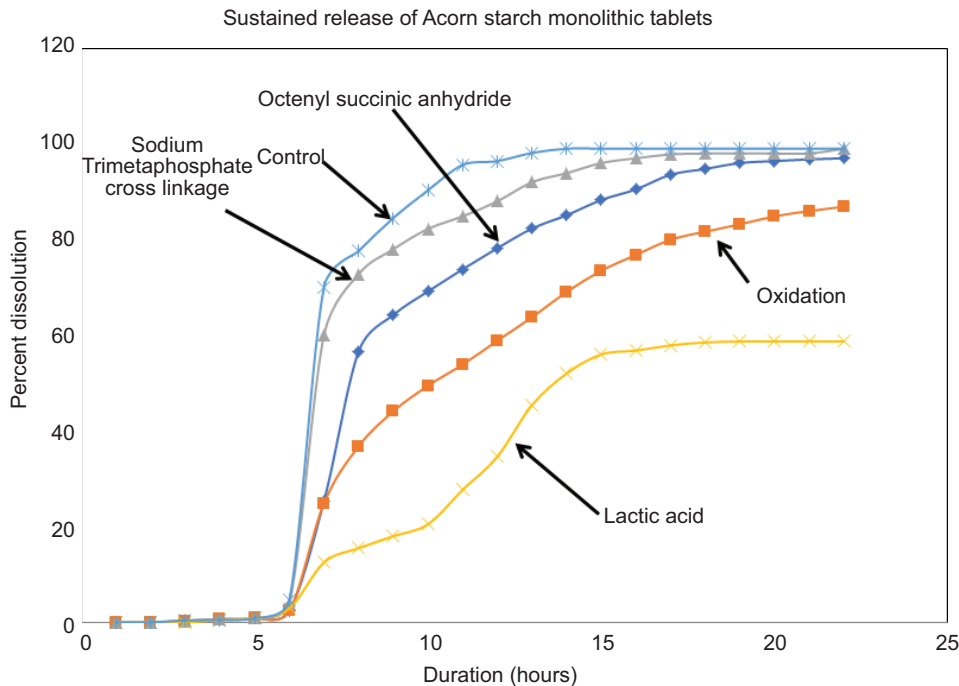


Figure 1. Sustained release (i.e., percent dissolution) of monolithic tablets made using modified (i.e., sodium trimetaphosphate cross-linkage, octenyl succinic anhydride, oxidation, and lactic acid) and nonmodified Acorn starch.

a more uniform structure, which may contribute to more predictable and extended-release characteristics (Leganes *et al.*, 2022).

Conclusions

The findings of this study highlight the significant impact of the type and degree of chemical modifications on the physicochemical properties of Acorn starch. Notably, modifications utilizing octenyl succinic anhydride and lactic acid enhanced the pasting viscosities and swelling capacities of the starch, excluding the temperature of 60°C, thus improving its functionality as a thickening agent in culinary contexts. The capability to modify starch characteristics through chemical means presents numerous opportunities for the optimization of starch-based materials tailored for specific industrial applications. Acorn modifications impacted additional functional properties, including gelatinization temperature, freeze–thaw stability, and retrogradation behavior, thereby enabling the precise enhancement of starch effectiveness in alignment with specific application needs. This adaptability positions chemically altered Acorn starch as a vital element across various industries, improving product quality and performance while offering economically feasible and sustainable alternatives.

Acknowledgments

The authors would like to thank the Deanship of Scientific Research, The University of Jordan for providing the financial support of this project (2466).

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Informed Consent

Written informed consent was obtained from all study participants.

Ethics Statement

The collection of the plants used in the study complies with local or national guidelines with no need for further affirmation. Ethical scrutiny and endorsement were unnecessary for the examination involving human subjects, aligning with local laws and institutional criteria.

Authors Contribution

Mohammad Saleh: Conceptualization, project supervision, data analysis, and manuscript preparation. Khaled Ismail: experimental design, methodology development, and manuscript writing. Ghadeer Mehyar: experimental analysis, application assessment, and manuscript writing and review. Radwan Ajo: Methodology development, experimental investigation, and manuscript drafting. Youngseung Lee: Experimental support, application analysis, and manuscript writing, review and editing.

Conflicts of Interest

The authors declare that they do not have any conflict of interest.

Funding

Project number 2466: The effects of chemically modified Acorn starch on syrup functionality and the sustained release properties of monolithic tablets. Deanship of Scientific Research, The University of Jordan, Amman, Jordan.

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