# Viscometric Characteristics and Physicochemical Properties of Acid-Modified Cocoyam Starch

Ghaferah H. Al-Hazmi,[a] Abimbola Aina Ogundiran,[b] Oyesolape Basirat Akinsipo,[b] Lamia A. Albedair,[a] Ali El-Rayyes,[c] Moamen S. Refat,[d] Kholoud K. Alzahrani,[e] and Akeem Adesina Bamigbade [f]

Cocoyam (*Colocasia* spp.) tuber, which has high starch yield, was subjected to acid modification. Effects were evaluated relative to the pasting, structural, physicochemical, and morphological properties. Native cocoyam starch (NCS) was treated with hydrochloric acid to produce acid-modified cocoyam starch (AMCS). Pasting properties were examined using a Rapid Visco Analyzer (RVA), while water and oil absorption capacities, swelling power, and gelatinization features were evaluated using standard methods. Morphological and structural alterations were analyzed. Acid treatment significantly higher peak, final, trough, and setback viscosities, signifying greater paste stability at high temperature, higher swelling capacity, and greater retrogradation tendency. AMCS also displayed higher gel-forming ability at lower concentrations, higher swelling power, and increased water and oil absorption capacities relative to NCS. Scanning electron microscopic images showed surface erosion and granule disruption after acid modification, while FT-IR spectra affirmed that modification occurred primarily *via* depolymerization without the introduction of new functional groups. X-ray diffraction analysis revealed retention of crystalline polymorphism with improved relative crystallinity after acid hydrolysis. In all, the acid treatment effectively altered the granular and molecular structure of cocoyam starch, enhancing its functional properties and demonstrating its suitability for food and industrial applications requiring strong gels, stable pastes, and enhanced hydration characteristics.

*DOI: 10.15376/biores.21.2.3910-3930*

*Keywords: Acid modification; Cocoyam starch; Pasting properties; Physicochemical properties; Rapid visco analyzer*

*Contact information: a: Department of Chemistry, College of Science, Princess Nourah bint Abdulrahman University, P.O. Box 84428, Riyadh 11671, Saudi Arabia; b: Department of Chemical Sciences, Tai Solarin Federal University of Education, Ijagun, Ogun State, Nigeria; c: Center for Scientific Research and Entrepreneurship, Northern Border University, 73213, Arar, Saudi Arabia, d: Department of Chemistry, College of Science, Taif University, P.O. Box, 11099, Taif 21944, Saudi Arabia; e: Department of Biology, University College of Umluj, University of Tabuk, Umluj, Tabuk, Saudi Arabia; f: Department of Chemistry, Federal University of Agriculture, Abeokuta, Ogun State, Nigeria;*
** Corresponding author: abimbolaogundiran701@gmail.com*

## INTRODUCTION

Starch is one of the most abundant biomacromolecules in nature and represents a major contribution to the human diet, serving as an energy source as well as a vital raw material for various applications in the industry (Falua *et al*. 2022; Shoukat *et al*. 2025). It is primarily derived from plant storage organs including tubers, roots, seeds, and grains, and it plays a crucial role in food systems because of its gelling, thickening, stabilizing, and binding properties (Rashwan *et al*. 2024; Culqui-Arce *et al*. 2025). The rising industrial demand for starch has led to a rise in the search for alternative botanical sources, with special focus in regions where diversification is needed for economic development and food security or where traditional starch crops may not be readily available (Adewale *et al*. 2022; Carvalho *et al*. 2024; Palmero *et al*. 2025).

Cocoyam (*Colocasia* spp.) has gained growing interest among these underexploited resources as a promising starch-bearing crop with substantial industrial potential for broader utilization (Boakye *et al*. 2018; Otekunrin *et al*. 2021). It is widely cultivated in South America, West Africa, and some parts of Asia, where it primarily serves as a dietary staple for different people (Boakye *et al*. 2018; Otekunrin *et al*. 2021). Despite its adaptability to varied ecological circumstances and nutritional value, the crop remains secondary to more dominant tuber and root crops such as yam, potato, and cassava. One of the most valued characteristics of cocoyam is its relatively high starch yield, with starch contents in the range of 60% to 80% on a dry weight basis, depending on the age of harvest, environmental and species factors (Jacob and Adeleke 2019).

Starch derived from cocoyam is characterized by small granule size, often within the range of 1 to 8 μm, and is much smaller than that of starches from other sources such as potato or cassava (Ojo *et al*. 2023; Chamorro *et al*. 2025). These smaller granule sizes provide functional advantages that enable the starch obtained from cocoyam to be attractive for specialized industrial uses, including as a binder in pharmaceutical tablets, a thickener in food products requiring smooth textures, and a filler in biodegradable films (Ariwaodo *et al*. 2017; Fadeyibi *et al*. 2022).

Nevertheless, just like many other native starches, starch obtained from cocoyam exhibits intrinsic limitations such as low shear resistance, poor thermal stability, and low tolerance to acidic environments that limit its direct application in certain processes in the industry (Amaraweera *et al*. 2021). These characteristics are a result of the specific arrangement of amylose and amylopectin molecules as well as the semi-crystalline nature of starch granules within their structure (Compart *et al*. 2023; Puri *et al*. 2025). For industries including food manufacturing, pharmaceuticals, paper production, and textiles, starch must often endure mechanical stress, varying pH conditions, and rapid heating and cooling cycles (Amaraweera *et al*. 2021; Puri *et al*. 2025). Thus, modifying native starch to enhance its functionality, adaptability, and stability has become a cornerstone of starch research and application (Puri *et al*. 2025).

Chemical treatment is one of the commonly used methods for altering starch characteristics. Among the numerous chemical treatment techniques such as oxidation, cross-linking, acid hydrolysis, and esterification, acid treatment is particularly valued for its low cost, predictable impact on starch structure, and simplicity (He *et al.* 2023). Acid treatment involves reacting starch granules with diluted mineral acids such as sulfuric acid, nitric acid, and hydrochloric acid, which selectively leave the crystalline zones relatively intact, but the reaction hydrolyzes the amorphous regions within the starch granule (Amaraweera *et al.* 2021; Chakraborty *et al*. 2022).

The molecular weight of starch polymers decreases, as hydrolysis progresses, leading to substantial changes in gelatinization behavior, viscosity, paste clarity, and solubility (Li and Hu 2021). Acidic modification of starches characteristically exhibits increased cold-paste viscosity, lower hot-paste viscosity, enhanced gel strength, and improved film-forming capacity (Chakraborty *et al*. 2022). The modification by acid also tends to gelatinize the starch at lower temperatures when compared other native counterparts (Javadian *et al*. 2021). These properties afford acid-modified starches highly suitable for industrial applications such as textile sizing, where smooth pastes enable fabric finish; confectionery production, where low-viscosity starch slurries are needed; and pharmaceutical binding, where enhanced flow and compatibility properties are essential.

Despite all these many documented advantages of acid modification, not much research has focused specifically on how this treatment impacts cocoyam starch. Most existing reports have focused on other dominant starches, such as cassava, maize, and potato. Since starch characteristics strongly depend on botanical origin, it cannot be assumed that the effects of acid modification noted in one starch type will be applicable to another. With cocoyam starch possessing distinctive granule morphology, unique crystalline structure, and amylose–amylopectin ratio that may respond in a different way to chemical modification, the understanding on how acid hydrolysis changes the physicochemical and functional properties of starch derived from cocoyam is a vital step toward expanding its commercial relevance. Similar to most other tuber starches, cocoyam starch is made up of approximately 20 to 30% amylose and 70 to 80% amylopectin, although the exact ratio depends on maturity, cultivar, and environmental conditions (Boakye *et al*. 2018; Ojo *et al*. 2023). Amylose usually consists of mostly linear $\alpha$-(1→4)-linked glucan chains with molecular weights commonly reported on the order of $10^5$ to $10^6$ g/mol, whereas amylopectin is a highly branched macromolecule with molecular weights usually in the range of $10^7$ to $10^9$ g/mol.

In several developing countries, such as Nigeria, there is a need to identify and promote local crops that can benefit small-scale and large-scale industrial production. Cocoyam is advantageous due to its tolerance to shade, relatively short maturation time, and adaptability to marginal soils, making it suitable for sustainable cultivation. Thus, starch extraction and modification will add value. Farmers and processors can reduce dependence on imported industrial starches and increase income opportunities. Accordingly, the aim of this study was to provide a detailed examination of the pasting and physicochemical properties of cocoyam starch subjected to acid modification. The use of Rapid Visco Analyzer (RVA) technology allows precise, reproducible measurement of pasting properties, while physicochemical analysis offers insight into the underlying structural alterations responsible for observed functional shifts.

## RESULTS AND DISCUSSION

### Starch Collection and Extraction

Fresh cocoyam tubers were obtained from a local farm in Ifo, Ogun State of Nigeria. They were washed with running tap water thoroughly to remove dirt and debris. About 15 cocoyam tubers were peeled and sliced into small pieces to simplify further processing and thereafter blended using a mechanical grinder with distilled water to produce a uniform slurry. The slurry product obtained was filtered using a muslin cloth to separate the fibrous residue from the starch-containing filtrate. The filtrate was allowed to stand undisturbed for 24 h to enable proper sedimentation of the starch and thereafter, the supernatant was carefully decanted, leaving behind the starch-rich sediment. The obtained sedimented starch was washed several times with distilled water to ensure the complete removal of soluble materials and residual impurities. The purified starch was oven dried at 45 °C, milled into powder, and referred to as native cocoyam starch (NCS).

## Acid Modification of Starch

About 100 g of NCS was suspended in a 1000 mL of 0.1 M hydrochloric acid, and the mixture was stirred continuously at room temperature using a magnetic stirrer (IKA C-MAG HS 7, IKA® Works, Germany) to ensure uniform contact between the acid and the starch granules. This stirring procedure was maintained for 12 h, after which the starch suspension was neutralized using 1 M sodium hydroxide and washed with distilled water until the pH reached 7.0. The modified starch was dried at 45 °C in an oven, ground to powder, and stored in airtight containers to prevent moisture absorption and referred to as acid-modified cocoyam starch (AMCS). The acid-modification conditions that was used in this study (0.1 M HCl, 12 h, room temperature) are comparable to mild acid-thinning treatments commonly reported for starch processing. In industrial practice, acid hydrolysis of starch is usually performed using dilute mineral acids (generally 0.05 to 0.2 M) under controlled time–temperature conditions to achieve partial depolymerization while maintaining granule integrity. The relatively moderate conditions selected in the present work were intended to simulate controlled acid thinning and to preferentially hydrolyze amorphous regions without causing extensive destruction of the crystalline framework.

## Water Solubility Index and Swelling Power Determination

Water solubility index (WSI) and swelling power (SP) were measured within the temperature range of 50 to 90 °C, at 10 °C intervals, following the modified procedure described by Jacob and Adeleke (2019). About 1 g of starch was weighed accurately and mixed with 50 mL of distilled water, and stirred gently for 10 min. Thereafter, the slurry formed was heated in a water bath at 50, 60, 70, 80, and 90 °C for 20 min, with intermittent stirring to avert clumping. After heating, the sample was centrifuged at 2500 rpm for 10 min. The supernatant was removed, and the sediment was weighed. The moisture content of the gel was then measured to estimate its dry matter content using the expressions below:

$$\text{Solubility (\%)} = \frac{\text{Weight of dissolved solids}}{\text{Original dry sample weight}} \times 100 \qquad (1)$$

$$\text{Swelling Power (g/g)} = \frac{\text{Weight of wet sediment}}{\text{Weight of dry starch}} \qquad (2)$$

## Viscometric-based Profiling

The pasting behavior of cocoyam starch was evaluated using a Rapid Visco Analyzer (RVA-4, Newport scientific, Australia). A starch suspension containing 10% w/w dry starch was held at 30 °C for 1 min, then heated to 95 °C at 5 °C/min and kept at this temperature for 10 min. It was then cooled to 50 °C at the same rate and held for 3 min. Pasting properties like temperature (T), trough viscosity (TV), viscosity (V), peak time (Pt), and final viscosity (FV) were measured. Breakdown viscosity (BV) was estimated as the difference between pasting viscosity (V) and trough viscosity (TV).

## Water and Oil Absorption Capacities of Native and Acid-Modified Starch Determination

Water and oil absorption capacities were determined by adopting a modified procedure described by Jung *et al*. (2017) and Jacob and Adeleke (2019). A 3 g portion of cocoyam starch was reacted with 40 mL of distilled water or oil and centrifuged for 10 min at 3500 rpm. Afterward, the mixture was centrifuged, the supernatant was decanted, and the tube was allowed to drain for an additional 15 min. The tube weight and its contents were then recorded, and the amount of bound water was estimated from the change in weight which was estimated using the expression below:

*Water Absorption Capacity (WAC):*

$$\text{WAC (g/g)} = \frac{\text{Weight of absorbed water}}{\text{Weight of dry sample}} \tag{3}$$

*Oil Absorption Capacity (OAC):*

$$\text{OAC (g/g)} = \frac{\text{Weight of absorbed oil}}{\text{Weight of dry sample}} \tag{4}$$

## Gelatinization Studies

Gelatinization was determined using the method of Lin *et al*. (2017). Starch samples in the concentrations range of 2% to 12% (w/v) were prepared in test tubes with 10 mL of distilled water. The suspensions were then stirred for 5 min, followed by heating in a water bath for 30 min at 80 °C. After heating, the tubes were rapidly cooled under running cold water and then chilled for 2 h at 4 °C.

## Fourier Transform Infrared Spectroscopy

Fourier transform infrared (FT-IR) spectroscopy was used to investigate the functional groups available in the starch powder before and after acid modification, using a Tensor 27 FT-IR spectrometer (Germany). The KBr pellet approach was adopted, in which 1.99% starch powder was mixed with KBr using a mortar and pestle and then compressed into a 2-mm-diameter pellet. Spectra were recorded from 4000 to 400 $\text{cm}^{-1}$ with a resolution of 4 $\text{cm}^{-1}$ and 64 scans.

## Scanning Electron Microscopy

The morphology and elemental composition of the native starch and its modified form were analyzed using a Zeiss EVO 18 scanning electron microscope (Carl Zeiss, Germany) that is equipped with energy-dispersive X-ray (EDX) spectroscopy. The instrument was operated under high-vacuum conditions at an accelerating voltage of 15 kV. Before the analysis, samples were mounted on carbon tape and sputter-coated with a thin layer of gold.

## X-Ray Diffraction

The crystalline structure of the native starch and its modified form were investigated by X-ray diffraction (XRD) using a Bruker D8 Advance diffractometer (Germany) equipped with Cu–Kα radiation ($\lambda$ = 1.5406 Å). The instrument was operated at 30 mA and 40 kV, and diffraction patterns were recorded at a scanning rate of 2°/min over a $2\theta$ range of 20° to 80°.

## Statistical Analysis

All measurements were carried out in triplicate and the average value reported. Data were analyzed using descriptive statistics and ANOVA to determine significant differences among treatments ($P < 0.05$).

## RESULTS AND DISCUSSION

### Pasting Properties

The pasting characteristics of NCS and AMCS, as evaluated by RVA, are represented in Table 1. The data were obtained from Figs. 1 and 2, respectively. It was observed that the acid treatment significantly influenced the viscosity properties of cocoyam starch, as exhibited by the increased peak viscosity of modified cocoyam starch (AMCS, 4413 RVU) compared to native cocoyam starch (NCS, 3995 RVU). The rise in peak viscosity seen in AMCS suggests that acid modification partially disrupted the granular structure, most especially in the amorphous regions, facilitating increased water penetration and granule expansion before rupture. This structural adjustment is consistent with observations in other chemically modified starches, where controlled substitution or hydrolysis creates new sites for hydration, thus enhancing the granule's capacity to swell and contribute to higher paste viscosities during RVA analysis (Saleh *et al*. 2025). Some chemical modifications such as esterification with octenyl succinic anhydride (OSA) have been reported to increase peak viscosity in modified starches relative to native counterparts, which is accredited to improved granule swelling and water interaction resulting from the introduced functional groups (Saleh *et al*. 2025). Controlled acid hydrolysis is known to specifically cleave glycosidic linkages in amorphous starch regions, which can enhance the accessibility of water by the molecules and facilitate swelling under thermal conditions (Compart *et al*. 2023).

Some literature reports highlight the fact that the impact of acid modification on pasting properties can vary with the type of acid used, conditions applied and concentration. There have also been some reports on reduced peak viscosities upon acid-treatment when depolymerization is extensive, leading to lower molecular weight distributions that cannot sustain high viscosity and swelling (Halim *et al*. 2024). This highlights that granule integrity and the degree of hydrolysis are vital determinants of the pasting properties, with moderate modification increasing viscosity and swelling, whereas severe hydrolysis lends to decline in these properties due to excessive chain cleavage. Thus, the higher peak viscosity of AMCS in this study indicates that acid treatment successfully altered granule structure in a way that enhanced water uptake and swelling, without extensive degradation of crystalline regions that would otherwise reduce pasting performance.

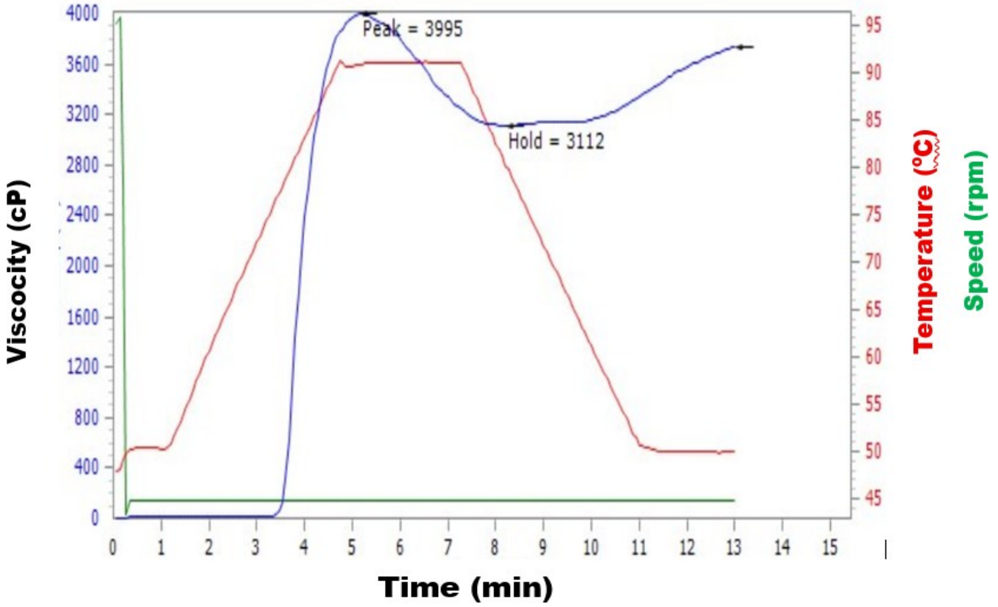


**Fig. 1.** RVA plot of native cocoyam starch

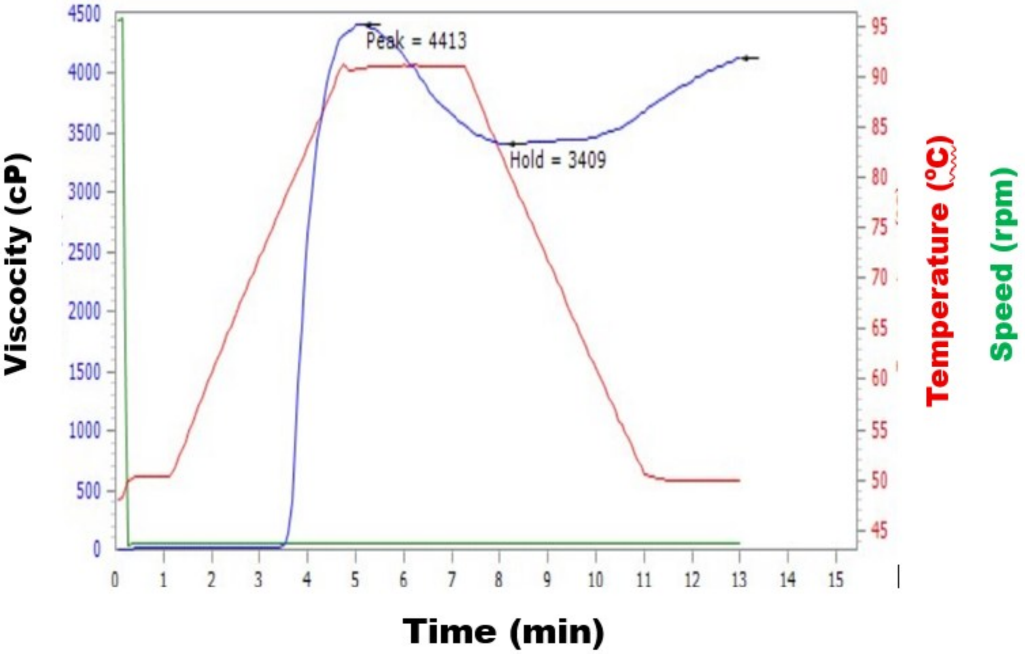


**Fig. 2.** RVA plot of acid-modified cocoyam starch

The pasting profiles of NCS and AMCS revealed clear differences in breakdown and trough viscosities, indicating alterations in paste stability during shear and heating. Trough viscosity (TV), which represents the minimum viscosity attained during prolonged high-temperature holding and shear conditions, was higher in AMCS (3409 RVU) compared to NCS (3112 RVU). An elevated TV indicates that the acid-treated starch paste demonstrated higher resistance to mechanical and thermal deformation resulting from gelatinization. In other words, the AMCS paste retained a comparatively greater structural integrity under sustained stirring and heat, which can be interpreted as enhanced paste stability during the holding phase. Though a majority of studies on acid treatment have focused on peak viscosity changes, conventional RVA interpretation acknowledges that a high trough value correlates with better viscosity retention during continuous heating, most especially in modified starches with altered molecular interactions or structural reorganization (Halim *et al.* 2025).

Notwithstanding this apparent enhancement in paste stability, the breakdown viscosity—computed as the difference between peak and trough viscosity—was also greater for AMCS (1004 RVU) than NCS (883 RVU). Breakdown viscosity is often accepted as an indicator of the susceptibility of swollen starch granules to breakdown under combined mechanical and thermal stress. A higher breakdown indicates that, after reaching maximum swelling, the granules are more readily disrupted and more susceptible to heat and shear. This thought is consistent with the known effects of acid hydrolysis on starch structure: acid modification specially cleaves glycosidic bonds in the amorphous regions of starch granules, causing partial depolymerization and a decline in molecular weight. These structural adjustments can weaken granular integrity, making the swollen granules more vulnerable to disintegration during the holding phase of pasting (Halim *et al.* 2025).

The combination of higher breakdown and trough viscosities in AMCS signify that while the treated starch can better retain viscosity at high temperature (higher trough), it also undergoes greater structural deformation once maximal swelling has occurred (higher breakdown) (Compart *et al*. 2023; Halim *et al*. 2024). These dual characteristics may suggest that acid treatment to a network can retain viscosity more effectively after gelatinization but is composed of smaller or less cohesive molecular fragments that are more prone to shear-induced breakdown (Compart *et al*. 2023; Halim *et al*. 2024). Related conceptual interpretations have been documented in starch pasting literature, where treatments that change molecular architecture (including acid hydrolysis) also at same time simultaneously influenced paste stability and granular resilience during continuous heating (Halim *et al.* 2025).

The final viscosity (FV) of a starch paste, evaluated at the end of the RVA cooling cycle, signifies the capability of starch molecules, most especially amylose and amylopectin, to reassociate and form a three-dimensional viscous network as the paste cools from the temperatures of the gelatinization. In this study, AMCS demonstrated greater final viscosity (4125 RVU) compared to NCS (3730 RVU), indicating that the acid-treated starch had an improved capacity to form a viscous gel upon cooling. This characteristic is typically indicative of enhanced molecular interactions during retrogradation, where leached amylose and amylopectin chains are realigning and entangle to reinforce the paste matrix (Chien-Chun 2009; Gong *et al*. 2024)). Literature reports on starch pasting features highlight that final viscosity correlates with gel strength and the extent of molecular entanglement; as better FV values are often associated with stronger paste structures upon cooling, which are beneficial in applications requiring thick, stable gels like custards, sauces, and fillings.

Setback viscosity (SB), which is defined as the difference between FV and TV, is often used as a proxy for retrogradation tendency—the reassociation of gelatinized starch molecules during storage and cooling. In this study, AMCS also demonstrated a higher setback viscosity (716 RVU) when compared with NCS (618 RVU), signifying an enhanced propensity for retrogradation. This enhanced setback could be accredited to the generation of shorter amylose chains and other depolymerized fragments formed during acid hydrolysis (Falade and Ayetigbo 2017; Li and Hu 2021). Shorter amylose chains are known to reassociate more quickly and widely during cooling because of their higher mobility and accessibility, thus promoting stronger gel networks and higher setback values (Falade and Ayetigbo 2017; Li and Hu 2021). Studies on root/tuber starches have previously reported that amylose composition and chain length greatly influence setback behavior, with greater amylose or shorter amylose units tending to form higher molecular reassociation and retrogradation (Chien-Chun 2009).

While some like hydrothermal or dual treatments can decrease setback by disrupting molecular ordering, controlled acid treatment may improve retrogradation propensities if the treatment increases the relative proportion of amylose accessible for reassociation during cooling (Javadian *et al*. 2021: Ghalambor *et al*. 2022; He *et al*. 2023). Acid modification often acts on the amorphous regions of starch granules; breaking bonds associated with glycosidic linkage and producing shorter polymer fragments without entirely preventing gel network formation (Wang and Copeland 2015; Soler *et al*. 2020). These shorter chains can associate rapidly as temperature declines, facilitating increased setbacks and final viscosity values relative to native starch. Thus, the observed greater FV and SB in AMCS likely indicate structural changes caused by acid modification that favor gel formation upon cooling.

In industrial application, starches with higher final and setback viscosities can provide higher gel firmness and cold paste viscosity, which are required in food formulations requiring stability upon cooling and thickened textures (Boonkor *et al*. 2022; Gong *et al*. 2024; Saleh *et al*. 2025). The pasting profile of both NCS and AMCS indicated no significant differences in pasting temperature (78.95 *vs.* 78.05 °C) or peak time (5.13 min). This indicates that acid modification, under the conditions applied in this study, did not greatly change the gelatinization thermal stability or kinetics associated with the initial swelling and disruption of starch granules. Related findings have been documented, where chemical modifications caused minimal impact on peak time, and pasting temperature, implying that the primary energy requirement needed for gelatinization remained comparable to the native starch (Javadian *et al*. 2021; Li and Hu 2021; He *et al*. 2023). The lack of noticeable alteration can be ascribed to the fact that acid hydrolysis in this case may have mainly targeted amorphous regions or caused mild depolymerization without significantly disrupting crystalline regions that govern gelatinization onset (Wang and Copeland 2015; Soler *et al*. 2020; He *et al*. 2023). Studies on starch treatment have shown that modifications affecting branching patterns or molecular weight do not always modify pasting onset temperatures except they substantially alter granule architecture or crystalline (Javadian *et al*. 2021).

Additionally, many starch treatment studies have shown that the effect on peak time and pasting temperature depends on the type and severity of modification. For instance, dual modifications that combine acid hydrolysis with other chemical or physical treatments often lead to shorter peak time and reduced pasting temperature because of increased granule disruption and depolymerization (Ghalambor *et al*. 2022). Nevertheless, in systems where acid modification is moderate and does not severely weaken crystalline order or intermolecular bonding, the time and the pasting temperature to reach maximum viscosity can remain similar between native and treated starches, as was observed in this study. Furthermore, reports on other modified starches have shown that great changes in peak time and pasting temperature are more commonly related with alterations to amylose–amylopectin or crystalline structure interactions instead of mere hydrolytic cleavage in amorphous regions (Ghalambor *et al*. 2022; Compart *et al*. 2023). As such, the present result supports the view that acid treatment, at levels applied here, changed viscosity characteristics (as noted in peak, trough, and final viscosities) without considerably affecting the gelatinization pathway itself (Javadian *et al*. 2021).

**Table 1.** RVA Parameters of Native and Acid-Modified Cocoyam Starch

| Pasting Parameters | NCS | AMCS |
|---|---|---|
| Peak Viscosity (cP) | 3995 | 4413 |
| Hold/Trough Viscosity (cP) | 3112 | 3409 |
| Breakthrough Viscosity (cP) | 883 | 1004 |
| Final Viscosity (cP) | 3730 | 4125 |
| Setback Viscosity (cP) | 618 | 716 |
| Peak Time (min) | 5.13 | 5.13 |
| Pasting Time (min) | 77.95 | 78.05 |

## Study on Swelling Power of NCS and AMCS

The swelling power (SP) results for NCS and AMCS showed a clear rise in water absorption and granule expansion as temperature was increased from 50 to 90 °C (Table 2). This behavior is consistent with the fundamental mechanism of starch gelatinization, where elevated temperature disrupts intermolecular hydrogen bonds, promoting molecular mobility, and improves the penetration of water into both crystalline and amorphous regions of the granule (Wang and Copeland 2015). When subjected to heating, the granule structure of the starch loosens, permitting more water uptake and subsequently greater swelling power values at higher temperatures (Wang and Copeland 2015). Within the range examined in this study, AMCS demonstrated higher swelling power when compared to NCS, particularly at elevated temperatures (*e.g.*, 2.08 g/g compared to 1.35 g/g at 90 °C). This improvement signifies that acid modification causes alterations the internal structure of cocoyam starch granules in a way that facilitates water absorption and expansion. While some studies often report declined swelling because of excessive hydrolysis or increased crystalline order, mild to moderate acid treatment can instead produce a more open granule matrix that enables water penetration and swelling under heat (Wang and Copeland 2015).

These reports are in alignment with recent observations in the literature, where dual modifications using acid hydrolysis (especially when combined with other treatments) caused a rise in swelling power at certain temperatures. For instance, dual modification strategies (acid hydrolysis plus ultrasonic treatment) have been reported to show an increase swelling power of cassava and similar starches at moderate temperatures, indicating improved water accessibility and structural loosening (Dewi *et al*. 2025). The noticeable increase in swelling power at elevated temperatures (≥80 °C) for AMCS emphasizes the importance of thermal energy in overcoming granule structural constraints. As temperature rises, intramolecular hydrogen bonds break, and the granule matrix become more amenable to expansion. A study conducted by Wang *et al.* (2015) indicated that temperature is a leading driver of swelling performance, regardless of modification, but that chemical modifications can temper with the magnitude of this response by causing alteration in the internal granule architecture (Wang *et al.* 2015). It is worth noting, however, that decreased swelling power after acid hydrolysis when conditions are harsher or when combined with treatments such as heat-moisture treatment has been reported in some research, which can reinforce intragranular bonding and resist expansion (Sun *et al*. 2015).

**Table 2.** Swelling Power of NCS and AMCS at Various Temperatures

| Samples | 50 ºC | 60 ºC | 70 ºC | 80 ºC | 90 ºC |
|---|---|---|---|---|---|
| SP for NCS (g/g) | 0.33 | 0.56 | 0.89 | 1.13 | 1.35 |
| SP for AMCS (g/g) | 0.45 | 0.88 | 1.04 | 1.63 | 2.08 |

## Water and Oil Absorption Capacities of Native and Acid-Modified Starch

The water absorption capacity (WAC) and oil absorption capacity (OAC) of native maize starch and its acid-modified counterpart were evidently different, indicating that chemical treatment changed the functional characteristics of starch (Table 3). The WAC of acid-modified starch (2.31 g/g) was considerably greater than that of raw maize starch (1.22 g/g), signifying improved hydration potency following acetylation. Similarly, OAC increased greatly after modification (from 0.81 to 1.75 g/g), signifying enhanced capacity to bind lipids. The rise in WAC upon acetylation is in alignment with recent reports that chemical modifications that introduce bulky substituents such as acetyl groups decrease the compactness of starch granules, thus increasing the accessibility of hydrophilic sites for water interaction (Suri and Singh 2023; Zhang *et al*. 2024).

Acetylation interrupts intramolecular hydrogen bonds and introduces steric hindrance, which facilitates hydration and water absorption across a broader range of starch structures (Chakraborty *et al*. 2022; Suri and Singh 2023). This behavior has been noticed in some legumes and tuber-based starches, where acetylated derivatives demonstrate great improvement in water absorption relative to native forms and such changes enhance thickening potential, textural properties, and water binding in food systems (Suri and Singh 2023). Directional modification, such as acetylation, mostly increases WAC by exposing additional hydroxyl groups and loosening the granule matrix, allowing improved starch–water associations and such enhancement of WAC can improve gel strength, viscosity, and moisture retention in foods where hydration is critical (Zhang *et al*. 2024).

Chemical modifications, such as acetylation, increase surface area and introduce structural irregularities, thus exposing hydrophobic sites capable of interacting with lipid molecules. Such enhanced OAC has been reported in studies comparing native with modified starches, supporting the notion that increased surface interaction sites and structural loosening facilitate oil binding (Suri and Singh 2023). A study on different native and modified starches (Adewumi *et al.* 2020) indicated that starches subjected to acetylation often demonstrated higher water and oil absorption capacities when compare with their native counterparts, signifying enhanced interfacial interactions with both polar (water) and non-polar (oil) phases and this kind of dual enhancement makes acetylated starches valuable as multifunctional ingredients, most importantly in complex food matrices in need of both lipid and hydration retention functionalities (Adewumi *et al.* 2020).

**Table 3.** Water and Oil Absorption Capacities of NCS and AMCS

| Parameters | NCS (g/g) | AMCS (g/g) |
|---|---|---|
| Water Absorption capacity (WAC) | 1.22 | 2.31 |
| Oil Absorption Capacity (OAC) | 0.81 | 1.75 |

## Gelatinization and Gelation of Native and Acid-Modified Cocoyam Starch in Relation to Recent Studies

The gelatinization and gelation properties of NCS and AMCS across concentrations (2% to 12% w/v) revealed clear functional differences, which is in alignment with reported effects of chemical treatments on starch gel structure (see Table 4). In both NCS and AMCS, inadequate network formation at low concentrations (2% to 4% w/v) indicated limited entanglement of amylose and amylopectin chains required for gel formation. It is known that higher polymer concentration elevates the likelihood of intermolecular interactions and junction zones that are vital for three-dimensional gel networks to produce granules gelatinize, and amylose leaches out when subjected to heating (Gong *et al*. 2024). For NCS, viscous features without a stable gel at 6% w/v and the first appearance of gel formation at 8% w/v signify that a minimum structural matrix of swollen granules and leached amylose is needed before a coherent gel network can sustain its shape upon cooling. On the other hand, AMCS already demonstrated evidence of gelation at 6% w/v and formed firmer gels at equivalent concentrations relative to NCS, resulting in a very firm gel at 12% w/v. This increased gel-forming capacity of acid-treated starch indicates that acid modification changed the molecular architecture of starch in a way that facilitated gel network development at lower concentrations or yielded stronger junction zones (Falade and Ayetigbo 2017; Gong *et al*. 2024).

Although the detailed review of starch gel structure mechanisms highlights multiple modification routes, acid hydrolysis precisely introduces shorter polymer chains and interrupts granule integrity, which can promote the leaching of amylose during gelatinization and improved gel network development (Gong *et al*. 2024). Moreover, studies on modified starches in other systems indicate that treatments can both increase and decrease least gelation concentration (LGC) depending on the severity and nature of modification. For instance, acid hydrolysis of yam and maize starches has been ascribed to enhanced gelling capacity and reduced minimum concentrations needed for gelation compared to native starch, supporting the present observation that AMCS begins to show structured gel properties at 6% to 8% w/v (Falade and Ayetigbo 2017). A recent study by He *et al*. (2023) observed structural insights indicating that acid modifications degrade certain starch polymer chains while leaving enough structural backbone to support entrenched network development upon cooling.

**Table 4.** Gelatinization Analysis of NCS and AMCS

| | | Sample Concentration (w/v) | | | | | |
|---|---|---|---|---|---|---|---|
| **Samples** | **Remark** | **2%** | **4%** | **6%** | **8%** | **10%** | **12%** |
| NCS | **State/ Gelation** | Liquid (No) | Liquid (No) | Liquid (No) | Viscous (Yes) | Least Gel Concentration (Yes) | Firm Gel (Yes) |
| AMCS | **State/ Gelation** | Liquid (No) | Liquid (No) | Viscous (Yes) | Least Gel Concentration (Yes) | Firm Gel (Yes) | Very Firm Gel (Yes) |

## Characterizations

The characterization results are illustrated in Figs. 3 to 6 and reveal that acid treatment of cocoyam starch induces coordinated morphological, chemical, and structural changes including oxygen-rich surfaces (EDS), eroded and porous granules (SEM), reorganized hydrogen bonding (FT-IR), and increased relative crystallinity (XRD). These changes provide mechanistic explanations for the improved swelling power, pasting performance, gel strength, and water absorption as noticed for AMCS.

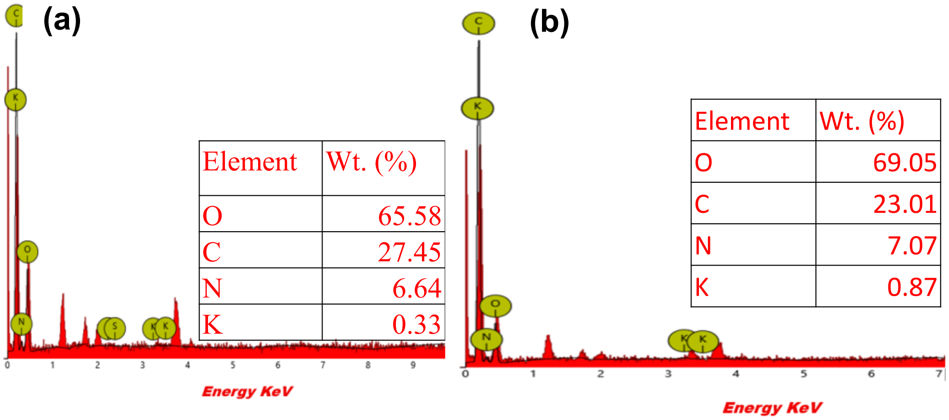
### *Energy-dispersive X-ray spectroscopy*

The surface elemental compositions of raw cocoyam starch and acid-modified cocoyam starch are seen in Figs. 3a and 3b. In the NCS, oxygen exhibited a high content (65.6 wt%), consistent with a rich presence of hydroxyl and glycosidic oxygen functionalities inherent to starch molecules. Carbon was also found at the level of 27.4 wt%, suggesting polysaccharide materials dominated by carbon backbone structures of amylose and amylopectin (Wang and Copeland 2015; Ojo *et al*. 2023). Minor elements such as nitrogen (6.6 wt%), and potassium (0.33 wt%) likely indicate the residual protein and mineral ions related to natural cocoyam starch granules and trace impurities often observed in root-derived starches.

Following acid modification, considerable shifts in elemental composition were noticed. The oxygen increased significantly to 69.0 wt%, whereas carbon content declined to 23.0 wt%. This relative rise in oxygen content signifies greater exposure or formation of oxygen-rich functional groups on the starch surface after acid modification. This can be potentially ascribed to partial hydrolysis of glycosidic bonds and increased surface accessibility of hydroxyl groups. The amorphous regions within starch granules are specially attacked by the acid hydrolysis, thereby causing a reduction in the molecular weight and exposing previously internal oxygen-bearing sites (Wang *et al*. 2019). In view of Xie *et al*. (2015), acid hydrolysis mostly erodes amorphous regions of starch, leaving behind more ordered crystalline structures and leading to an increase in crystallinity.

The reduction in carbon percentage observed may also indicate a relative loss of carbon-rich organic matter, as hydrolytic cleavage generates smaller oligosaccharides that may be washed away during activation. Nitrogen content rose to 7.07 wt% post-activation, which may have been from enrichment or concentration of proteinaceous residues on the granule surface after partial removal of carbohydrate fractions during acid modification, a characteristic occasionally seen when non-carbohydrate components remain bound to modified starch matrices (Wang and Copeland 2015; He *et al*. 2023).

Collectively, the EDS results showed that acid modification created a more inorganic-lean and oxygen-rich surface when compared with native starch. Such chemical modifications are in alignment with reported effects of acid hydrolysis on starch structure, where protonation of glycosidic oxygen leads to the disintegration of amorphous domains and increased relative crystallinity as the more ordered regions become predominant (Chakraborty *et al*. 2022; Zhang *et al*. 2023). This increased exposure of oxygen functional groups and altered mineral content could improve functional and reactivity performance of acid-treated starches in applications requiring high solubility, surface polarity, or interaction with other chemical species, such as in composite materials, adsorbents, or food formulations.



(a)

| Element | Wt. (%) |
|---|---|
| O | 65.58 |
| C | 27.45 |
| N | 6.64 |
| K | 0.33 |

(b)

| Element | Wt. (%) |
|---|---|
| O | 69.05 |
| C | 23.01 |
| N | 7.07 |
| K | 0.87 |

**Fig. 3.** EDS Analysis of (a) Native; and (b) Acid-modified cocoyam starch

*Scanning electron microscopic analysis*

Figure 4 displays the morphological differences between native and acid-modified cocoyam starch granules. The NCS granules showed relatively compact structures, smooth surfaces, and well-defined shapes, which are in alignment with previous reports describing cocoyam starch as having small, polygonal to rounded granules with intact surfaces (Ariwaodo *et al*. 2017; Ojo *et al*. 2023). On the other hand, AMCS showed visible surface erosion, fissures, roughness, and occasional pitting. These morphological interruptions are features of acid hydrolysis, which selectively attacks amorphous regions while leaving crystalline lamellae relatively intact. Surface weakening and internal stress result in granule cracking and pore formation as hydrolysis progresses.

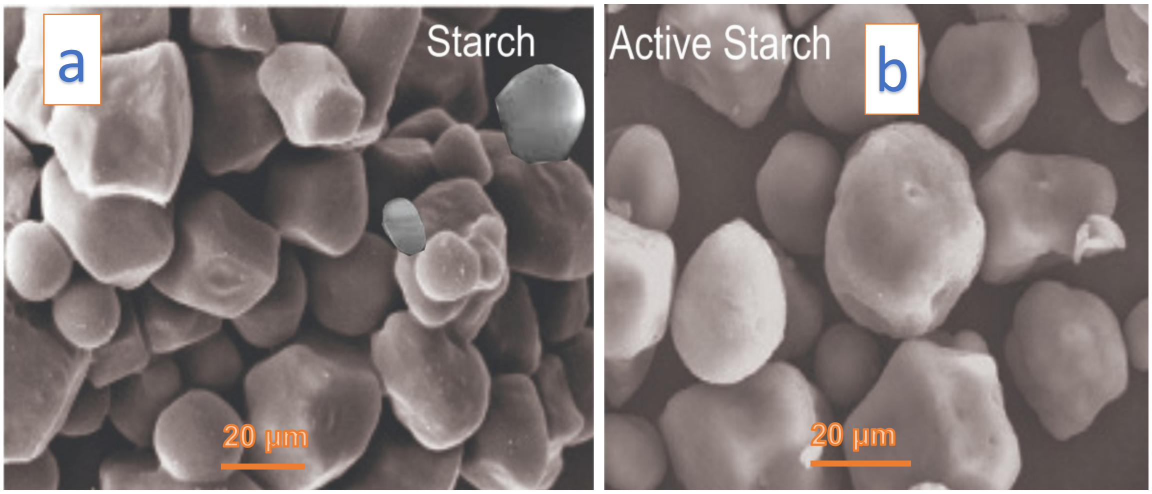


**Fig. 4.** SEM Analysis of (a) Native; and (b) Acid-modified cocoyam starch

Similar SEM features have been documented for acid-treated maize, cassava, yam, and quinoa starches, where partial fragmentation and surface roughening reflect controlled depolymerization (Chakraborty *et al*. 2022; Halim *et al*. 2024). The increased surface irregularity noticed in AMCS correlates well with its increased WAC and SP, supporting the observed improvements in pasting and hydration properties of AMCS, as surface defects enable water penetration into the granule interior and accelerating hydration and gelatinization under thermal conditions.

## Fourier Transform Infrared Spectroscopy

Figure 5 displays the FT-IR spectra of NCS and AMCS, indicating the chemical integrity of the starch before and after acid treatment. Both spectra exhibit features of starch absorbance bands with broad O–H stretching vibrations shown at 3255 to 3480 cm$^{-1}$, C–H stretching close to 2920 cm$^{-1}$, and strong C–O–C and C–O stretching peaks in the 1040 to 1150 cm$^{-1}$ region, which are related to glycosidic linkages (Lin *et al*. 2017; Soler *et al*. 2020; Zhang *et al*. 2023).

After acid treatment, no new functional group peaks were observed, suggesting that acid hydrolysis changed the starch mainly *via* depolymerization instead of chemical substitution (Wang and Copeland 2015; Soler *et al*. 2020; Li and Hu 2021). The rearrangement of hydroxyl groups and alterations of intermolecular interactions can be attributed to acid hydrolysis, which caused disruption of amorphous regions and is often reflected as changes in FT-IR band intensities instead of the appearance of new peaks (Soler *et al*. 2020; Li and Hu 2021). Recent research on acid-treated starches consistently reports that FTIR spectra retain core starch signatures while displaying subtle intensity variations related to the rise in crystallinity or reorganization of molecular domains (He *et al*. 2023; Zhang *et al*. 2023).

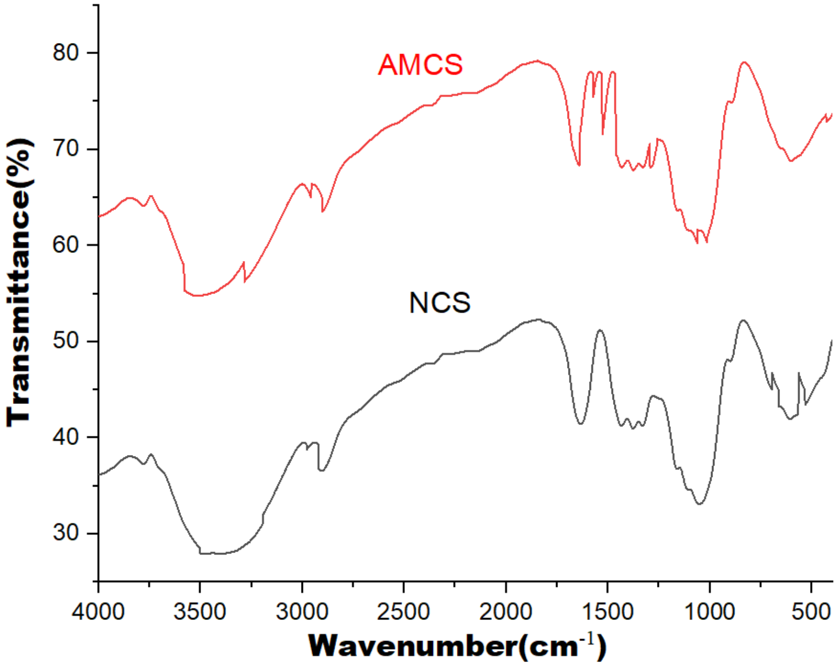


**Fig. 5.** FTIR Analysis of native cocoyam starch (NCS); and acid-modified cocoyam starch (AMCS)

## X-Ray Diffraction Analysis

Figure 6 shows the XRD patterns of NCS and AMCS with the native starch exhibiting diffraction peaks characteristic of A-type crystallinity, which is commonly seen in tropical root and tuber starches with relatively dense packing of double helices. The observed well-defined peaks suggest semi-crystalline granules made up of alternating crystalline and amorphous regions.

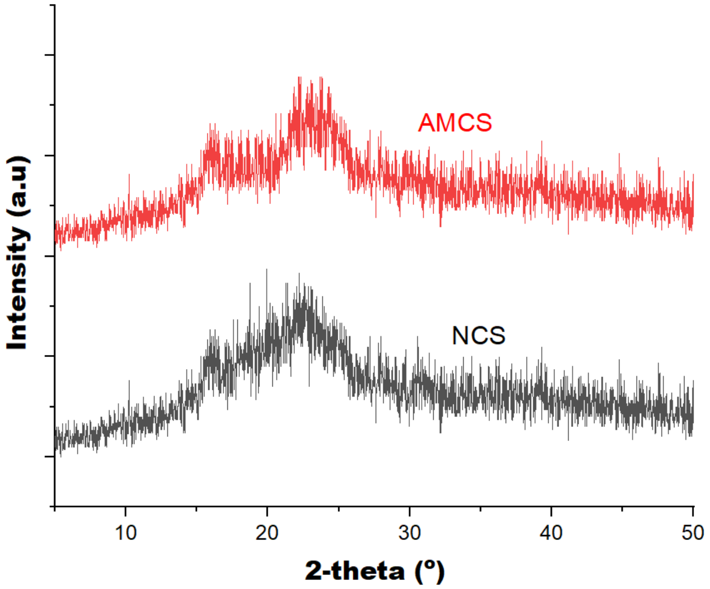


**Fig. 6.** XRD Analysis of native cocoyam starch (NCS) and acid-modified cocoyam starch (AMCS)

After acid treatment, the diffraction pattern retains the same polymorphic type but shows improved peak intensity and sharpness, signifying a relative increase in crystallinity. Similar trends have been documented in acid-hydrolyzed potato, maize, cocoyam, and cassava starches (Wang and Copeland 2015; Soler *et al*. 2020; Ojo *et al*. 2023). The improvement in relative crystallinity observed explains several functional changes seen in AMCS, such as enhanced gel firmness and higher final viscosity. While crystalline regions resist swelling, their enhancement following elimination of amorphous domains causes stronger molecular reassociation during cooling, thereby facilitating gel network formation and retrogradation. Some studies highlight that controlled acid hydrolysis can optimize the balance between amorphous flexibility and crystalline stability, producing starches with enhanced gel strength and cold-paste viscosity (Chakraborty *et al*. 2022; Gong *et al*. 2024). The observed changes in the XRD patterns can be interpreted in light of amylose crystallization behavior. During cooling and storage, leached amylose chains tend to reassociate into ordered single- and double-helical structures, which subsequently pack into crystalline domains. Amylose has been confirmed as the primary contributor to early retrogradation and gel network formation, leading to increased structural order detectable by XRD (Guo *et al*. 2024). In addition, amylose can organize into multiple aggregate forms such as V-type complexes and retrograded crystallites, each contributing differently to starch functionality and setback behavior (Shao *et al*. 2025). The progressive increase in helical order during retrogradation has been well documented, with amylose double helices forming rapidly and dominating early crystalline development (Lu *et al*. 2023).

## CONCLUSIONS

This study examined the modification of cocoyam starch using acid hydrolysis method. Results obtained from the study are summarized below:

1. Treatment with acid significantly improved the pasting properties of cocoyam starch, as evidenced by rheological properties compared to native starch.
2. Acid-modified cocoyam starch displayed higher swelling power, increased gel-forming capacity at lower concentrations, and greater water and oil absorption capacities, indicating enhanced hydration and network-forming ability.
3. The acid treatment induced surface erosion and granule disruption, signifying preferential degradation of amorphous regions while maintaining overall granule integrity as revealed by morphological analysis.
4. Selective amorphous-region hydrolysis was confirmed *via* X-ray diffraction analysis, which showed retention of the native crystalline polymorphic pattern with increased relative crystallinity, indicating enrichment of crystalline domains.
5. Investigation *via* Fourier transform infrared (FT-IR) spectroscopy revealed that acid treatment did not introduce new functional groups, signifying that structural changes occurred mainly through reorganization and depolymerization of hydrogen bonding rather than chemical substitution.
6. Conclusively, acid treatment proved to be an effective approach for improving its suitability for food, enhancing cocoyam starch functionality, and industrial applications.

## ACKNOWLEDGMENTS

Princess Nourah bint Abdulrahman University Researchers Supporting Project number (PNURSP2026R76), Princess Nourah bint Abdulrahman University, Riyadh, Saudi Arabia. Also, the authors extend their appreciation to the Deanship of Scientific Research at Northern Border University, Arar, KSA, for funding this research work through the project number “NBU-FFR-2026-2985”

### Authors Contributions

Ghaferah H. Al-Hazmi and Ali El-Rayyes: Contributed Resources, Software, Funding acquisition; Lamia A. Albedair: Project administration, Contributed Resources, Writing – Review & Editing; Abimbola Aina Ogundiran: Conceptualization, Investigation, and supervised the work; Moamen S. Refat: Visualization, Validation, Writing – Review & Editing; Kholoud K. Alzahrani: Formal analysis, Software, Writing – Review & Editing; Oyesolape Basirat Akinsipo and Akeem Adesina Bamigbade: Methodology, Data curation, Writing – Original Draft

## REFERENCES CITED

Adewale, P., Yancheshmeh, M. S., and Lam, E. (2022). "Starch modification for non-food, industrial applications: Market intelligence and critical review," *Carbohydr. Polym.* 291, article 119590. https://doi.org/10.1016/j.carbpol.2022.119590

Adewumi, F. D., Lajide, L., Adetuyi, A. O., and Ayodele, O. (2020). "Functional properties of three native starches and their modified derivatives," *Potravinarstvo Slovak Journal of Food Sciences* 14, 682-691. https://doi.org.10.5219/1232

Amaraweera, S. M., Gunathilake, C., Gunawardene, O. H. P., Fernando, N. M. L., Wanninayaka, D. B., Dassanayake, R. S., Rajapaksha, S. M., Manamperi, A., Fernando, C. A. N., Kulatunga, A. K., and Manipura, A. (2021). "Development of starch-based materials using current modification techniques and their applications: A review," *Molecules* 26(22), article 6880. https://doi.org.10.3390/molecules26226880

Ariwaodo, C. A.., Ezeama, C. F., and Nwabueze, T. U. (2017). "Morphology, rheology and functional properties of starch from cassava, sweet potato and cocoyam," *Asian J. Biol.* 3(3), 1-13. https://doi.org.10.9734/AJOB/2017/34587

Boakye, A. A, Wireko-Manu, F. D., Oduro, I., Ellis, W. O., Gudjónsdottir, M., and Chronakis, I. S. (2018). "Utilizing cocoyam (*Xanthosoma sagittifolium*) for food and nutrition security: A review," *Food Sci. Nutr*. 6(4), 703-713. https://doi.org/10.1002/fsn3.602

Boonkor, P., Sagis, L. M. C., and Lumdubwong, N. (2022). "Pasting and rheological properties of starch paste/gels in a sugar-acid system," *Foods* 11(24), article 4060. https://doi.org/10.3390/foods11244060

Carvalho, H. J. M., Barcia, M. T., and Schmiele, M. (2024). "Non-conventional starches: Properties and potential applications in food and non-food products," *Macromol* 4(4), 886-909. https://doi.org/10.3390/macromol4040052

Chakraborty, I. N. P., Mal, S. S., Paul, U. C., Md. Hafizur, R., and Mazumder, N. (2022). "An insight into the gelatinization properties influencing the modified starches used in food industry: A review," *Food Bioprocess Technol.* 15, 1195-1223. https://doi.org/10.1007/s11947-022-02761-z

Chamorro, A. F., Palencia, M., and Lerma, T. A. (2025). "Physicochemical characterization and properties of cassava starch: A review," *Polymers* 17(12), article 1663. https://doi.org/10.3390/polym17121663

Chien-Chun, H. (2009). "Physicochemical, pasting and thermal properties of tuber starches as modified by guar gum and locust bean gum," *International Journal of Food Science and Technology* 44(1), 50-57. https://doi.org/10.1111/j.1365-2621.2007.01634.x

Compart, J., Singhm A., Fettke, J., and Apriyanto, A. (2023). "Customizing starch properties: A review of starch modifications and their applications," *Polymers (Basel)* 15(16), article 3491. https://doi.org/10.3390/polym15163491

Culqui-Arce, C., Paucar-Menacho, L. M., Schmiele, M., Vito-Villa, J., Camacho-León, J., Mori-Mestanza, D., Auquiñivin-Silva, E. A., Cayo-Colca, I. S., Castro-Alayo, E. M., and Balcázar-Zumaeta, C. R. (2025). "Unveiling the potential of starch from tubers and grains as primary source of biopolymers," *Discov Food* 5, article 288. https://doi.org/10.1007/s44187-025-00598-x

Dewi, L. K., Athif, F. N., Sasikirana, A. N., Bayu, A. I., Panggau, A. K., Sugiono, R. S., and Sumardiono, S. (2025). "The effect of dual modification on swelling power and solubility of cassava starch modified by lactic acid hydrolysis and ultrasonication," *AIP Conf. Proc.* 3309, article 060002. https://doi.org.10.1063/5.0264980

Fadeyibi, A., Alabi, K. P., Fadeyibi, M., and Adewara, A. O. (2022). "Synthesis, characterization, and suitability of cocoyam starch-banana peels nanocomposite film for locust beans packaging," *Bull. Natl. Res. Cent.* 46, article 190. https://doi.org.10.1186/s42269-022-00882-1

Falade, K. O., and Ayetigbo, O. E. (2017). "Effects of tempering (annealing), acid hydrolysis, low-citric acid substitution on chemical and physicochemical properties of starches of four yam (*Dioscorea* spp.) cultivars," *J. Food Sci. Technol*. 54(6), 1455-1466. https://doi.org.10.1007/s13197-017-2568-x

Falua, K. J., Pokharel, A., Babaei-Ghazvini, A., Ai, Y., and Acharya, B. (2022). "Valorization of starch to biobased materials: A review," *Polymers* 14(11), article 2215. https://doi.org.10.3390/polym14112215

Ghalambor, P., Asadi, G., Mohammadi, N. A., and Seyedin, A. S. M. (2022). "Investigation of dual modification on physicochemical, morphological, thermal, pasting, and retrogradation characteristics of sago starch," *Food Sci Nutr.* 10(7), 2285-2299. https://doi.org.10.1002/fsn3.2837

Gong, Y., Xiao, S., Yao, Z., Deng, H., Chen, X., and Yang, T. (2024). "Factors and modification techniques enhancing starch gel structure and their applications in foods: A review," *Food Chemistry* X 24, 102045. https://doi.org.10.1016/j.fochx.2024.102045

Guo, K., Tian, Y., Podzimska-Sroka, D., Kirkensgaard, J. J. K., Herburger, K., Enemark-Rasmussen, K., Hassenkam, T., Petersen, B. L., Blennow, A., and Zhong, Y. (2024). "Structural evolution of maize starches with different amylose content during pasting and gelation as evidenced by Rapid Visco Analyser," *Food Chemistry* 461, article 140817. https://doi.org/10.1016/j.foodchem.2024.140817

Lu, H., Tian, Y., and Ma, R. (2023). "Assessment of order of helical structures of retrograded starch by Raman spectroscopy," *Food Hydrocolloids* 134, article 108064. https://doi.org/10.1016/j.foodhyd.2022.108064

Shao, C., Yu, J., Copeland, L, and Wang, S. (2025). "Association of natural amylose into different forms of aggregates: Structure and potential mechanisms," *J. Agric. Food Chem*.73(47), 30500-30510. DOI: 10.1021/acs.jafc.5c12555.

Halim, J. K., Aldama, S. G., Noviandari, A. F., Che Ku Jusoh, T. F. I., Utami, N. P., Hidayah, N., and Fitriani, A. (2025). "Formation of type III resistant starch and alterations in starch characteristics and pasting behavior of arrowroot starch by autoclaving-cooling treatment," *Curr. Appl. Sci. Technol.* 2025, article e0267208. https://doi.org.10.55003/cast.2025.267208

Halim, A., Torley, P. J., Farahnaky, A., and Majzoobi, M. (2024). "Investigating the effects of acid hydrolysis on physicochemical properties of quinoa and faba bean starches as compared to cassava starch," *Foods* 13(23), article 3885. https://doi.org.10.3390/foods13233885

He, R., Li, S., Zhao, G., Zhai, L., Qin, P., and Yang, L. (2023). "Starch modification with molecular transformation, physicochemical characteristics, and industrial usability: A state-of-the-art review," *Polymers* 15(13), article 2935. https://doi.org.10.3390/polym15132935

Jacob, O. A., and Adeleke, O. A. (2019). "Isolation and characterization of starch obtained from cocoyam cultivated at Akungba Akoko, Ondo State, Nigeria," *Nutri. Food Sci. Int J.* 8(2), article 555732. https://doi.org.10.19080/NFSIJ.2018.08.555732

Javadian, N., Mohammadi, N. A., and Bolandi, M. (2021). "The effects of dual modification on functional, microstructural, and thermal properties of tapioca starch," *Food Sci. Nutr*. 9(10), 5467-5476. https://doi.org.10.1002/fsn3.2506

Jung, Y.S., Lee, B.H., and Yoo, S.H. (2017). "Physical structure and absorption properties of tailor-made porous starch granules produced by selected amylolytic enzymes," *PLoS One.* 12(7), e0181372. https://10.1371/journal.pone.0181372

Li, C., and Hu, Y. (2021). "Effects of acid hydrolysis on the evolution of starch fine molecular structures and gelatinization properties," *Food Chemistry* 353*,* article 129449. https://doi.org.10.1016/j.foodchem.2021.129449

Lin, D., Zhou, W., Zhao, J., Lan, W., Chen, R., Li, Y., Xing, B., Li, Z., Xiao, M., Wu, Z., *et a*l. "Study on the synthesis and physicochemical properties of starch acetate with low substitution under microwave assistance," *International Journal of Biological Macromolecules* 103, 316-326. https://doi.org.10.1016/j.ijbiomac.2017.05.056

Ojo, T. P., Sanni, T. A., and Arinola S. O. (2023). "Effect of modifications on the chemical composition, X-ray diffraction pattern and scanning morphology of starches from cocoyam (*Colocasia esculenta* and *Xanthosoma sagittifolium*)," *Asian Food Sci. J.* 22 (8), 17-27. https://doi.org.0.9734/afsj/2023/v22i8650

Otekunrin, O. A., Sawicka, B., Adeyonu, A. G., Otekunrin, O. A., and Rachoń, L. (2021). "Cocoyam [*Colocasia esculenta* (L.) Schott]: Exploring the production, health and trade potentials in sub-Saharan Africa," *Sustainability* 13(8), article 4483. https://doi.org.10.3390/su13084483

Palmero, G. M., Carrasco, A., de la Sen, P., Mainar-Toledo, M. D., Malo, A. S., and Herrer, R. F. J. (2025). "Starch valorisation as biorefinery concept integrated by an agro-industry case study to improve sustainability," *Sustainability* 17(15), article 6808. https://doi.org.10.3390/su17156808

Puri, A., Mohite, P., Ramole, A., Verma, S., Kamble, M., Ranch, K., and Singh, S. (2025). "Starch science advancement: Isolation techniques, modification strategies, and multifaceted applications," *Macromol*. 5(3), article 40. https://doi.org.10.3390/macromol5030040

Rashwan, A. K., Younis, H. A., Abdelshafy, A. M., Osman, A. I., Eletmany, M. R., Hafouda, M. A., and Chen, W. (2024). "Plant starch extraction, modification, and green applications: A review," *Environ Chem Lett* 22, 2483-2530. https://doi.org.10.1007/s10311-024-01753-z

Saleh, M., Al-Ismail, K., Mehyar, G., Ajo, R., and Lee, Y. (2025). "Effects of chemically modified corn starch on its functional characteristics and the sustained release properties of monolithic tablets," *Italian Journal of Food Science* 37(3), 108-119. https://doi.org.10.15586/ijfs.v37i3.3001

Shoukat, R., Cappai, M., Pilia, L., and Pia, G. (2025). "Rice starch chemistry, functional properties, and industrial applications: A review," *Polymers* 17(1), article 110. https://doi.org.10.3390/polym17010110

Soler, A., Valenzuela-Díaz, E. D., Velazquez, G., Huerta-Ruelas, J. O., Morales-Sanchez , E., Hernandez-Gama, R., and Mendez-Montealv, G. (2020). "Double helical order and functional properties of acid-hydrolyzed maize starches with different amylose content," *Carbohydrate Research* 490, article 107956. https://doi.org.10.1016/j.carres.2020.107956

Sun, Q., Zhu, X., Si, F., and Xiong, L. (2015). "Effect of acid hydrolysis combined with heat moisture treatment on structure and physicochemical properties of corn starch," *J. Food Sci. Technol.* 52(1), 375-82. https://doi.org.10.1007/s13197-013-0998-7

Suri, S., and Singh, A. (2023). "Modification of starch by novel and traditional ways: influence on the structure and functional properties," *Sustainable Food Technol.* 1, 348-362. https://doi.org.10.1039/D2FB00043A

Wang, S., and Copeland, L. (2015). "Effect of acid hydrolysis on starch structure and functionality: A review," *Crit. Rev. Food Sci. Nutr.* 55(8), 1081-1097. https://doi.org.10.1080/10408398.2012.684551

Zhang, J., Tao, L., Yang, S., Li, Y., Wu, Q., Song, S., and Yu. L. (2024). "Water absorption behavior of starch: A review of its determination methods, influencing factors, directional modification, and food applications," *Trends in Food Science and Technology* 144, article 104321. https://doi.org.10.1016/j.tifs.2023.104321

Zhang, M., Jia, H., Wang, B., Ma, C., He, F., Fan, Q., and Liu, W. (2023). "A prospective review on the research progress of citric acid modified starch," *Foods* 12(3), article 458. https://doi.org.10.3390/foods12030458

Article submitted: January 24, 2026; Peer review completed: February 21, 2026; Revised version received: February 25, 2026; Accepted: February 26, 2026; Published: March 13, 2026.
DOI: 10.15376/biores.21.2.3910-3930