Physicochemical and Rapid Visco Analyzer Profiling of Oxidized Maize Starch

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The impact of an oxidation process was examined relative to the physicochemical, functional, and structural characteristics of maize starch. Raw maize starch (RMS) was extracted *via* wet milling, while oxidized maize starch (OMS) was produced through controlled oxidation. Gelation behavior, pasting characteristics using Rapid Visco Analyzer (RVA), water and oil absorption capacities, structural change analyses, and swelling power were examined. The oxidation process significantly increased the water and oil absorption capacities of (1.5±0.01 g/g and 1.2±0.02 g/g) in OMS compared with RMS (0.9±0.01 g/g and 0.6±0.01 g/g) respectively, signifying increased hydrophilicity and exposure of non-polar sites. RVA results exhibited reductions in trough, peak, final, and setback viscosities, suggesting decreased retrogradation tendency and enhanced paste stability under heat and wear. OMS showed improved swelling power across all temperatures (55 to 95 °C), indicating increased hydroxyl accessibility and disruption of crystalline regions. Fourier transform infra-red spectra exhibited shifts in carbonyl and hydroxyl functional groups, while scanning electron microscopy showed granule alteration and surface roughening. Overall, the oxidation process enhanced thermal, hydration, and structural properties of maize starch, signifying its potential for industrial applications requiring better solubility, reduced retrogradation, and improved paste stability.

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INTRODUCTION

Maize (*Zea mays*) is one of the most significant cereal crops globally, serving as a fundamental food source and a vital industrial raw material. Much maize is utilized for the purpose of animal feeds, both as grain and as the whole plant, such that it can either be made into the more palatable silage or baled. Sugar-rich varieties called sweet corn are grown for human consumption, while field corn varieties are used for animal feed, for uses such as masa or cornmeal, corn syrup, corn starch, alcoholic beverages such as bourbon whiskey, pressing into corn oil, and as chemical feedstocks including ethanol and other biofuels (Erenstein *et al*. 2022; Madhu 2022).

Key sources of starch include maize, wheat, and potatoes. As a natural polymer or polysaccharide, starch consists of long chains of glucose molecules. Maize starch is the most commercially important starch globally, accounting for a large portion of the world's total starch production (Niu *et al*. 2023; Wang and Lu 2023). Starch has a granular form with semi-crystalline structure found within the chloroplasts of green leaves and the amyloplasts of tubers and grains (Luo *et al*. 2020). Its functionality is vital for its numerous applications. Within the food industry, starch acts as a thickener, gelling agent, and stabilizer, influencing texture and mouthfeel (Watcharakitti *et al*. 2022; Gong *et al*. 2024). In industrial applications, starch serves as a binder, adhesive, and film-forming agent (Linan *et al.* 2024). Factors such as the distribution of branched chains in amylopectin, amount of amylose, granular architecture, and the presence of phospholipids, phosphate monoesters, and lipids greatly affect the functional properties of starch (Lu *et al*. 2024; Zhai *et al*. 2024).

Despite its versatility, native maize starch exhibits some limitations such as restricted swelling capacity, low solubility, and poor thermal stability. These constrain its functional applications and thus, capable of hindering its functionality in various applications, necessitating the need for starch modification techniques to enhance its properties and expand its industrial utility (Tarahi *et al*. 2022).

One of the prominent techniques for starch modification is chemical activation, which alters its physicochemical and functional properties (Suri and Singh 2023). This process typically results in changes to the granule structure, molecular weight, and functional attributes of the starch, making it more suitable for specific industrial applications (He *et al*. 2023).

Research has demonstrated that chemical activation can significantly improve the swelling power, solubility, and gelatinization properties of starch (Suri and Singh 2023). Chemical modification of starch can be achieved through various solvents, including hydrochloric acid, sulfuric acid, citric acid, hypochlorite or hydrogen peroxide, each inducing varying degrees of modification in the starch swelling power (He *et al*. 2023; Cavallo *et al.* 2024). Chemical activation induces notable changes in the physicochemical properties of starch, thereby enhancing its functionality for diverse applications (Zhang *et al*. 2023a). Chemical-activated starch stands out due to its ability to improve the starch solubility. In particular, changes are driven by the disruption of crystalline regions, which leads to the formation of smaller, more soluble fragments (Skendi *et al*. 2020).

Among the analytical methods employed to examine the functionality of starch is the Rapid Visco Analyzer (RVA), which has gained widespread interest as a rapid and reliable tool for investigating viscosity and pasting behavior under conditions that simulate thermal processing (Balet *et al*. 2019). The Rapid Visco Analyzer functions by subjecting starch suspensions to a controlled heating–cooling cycle under constant shear while continuously measuring viscosity as torque resistance (Balet *et al.* 2019). This enables real-time monitoring of crucial transitions like such as swelling, breakdown under shear, gelatinization, and molecular reassociation during cooling (Tian *et al*. 2022).

Compared with other rheological and viscometric techniques, the RVA gives distinct advantages in starch analysis. Though conventional rotational rheometers such as cone-and-plate or parallel plate systems are capable of evaluating fundamental rheological properties including viscoelastic moduli and yield stress; nevertheless, they usually require more complex experimental design and are less standardized for routine starch pasting evaluation (Nastasi *et al*. 2025).

Capillary viscometers, though useful for measuring intrinsic viscosity under laminar flow conditions, do not replicate the combined effects of heat and shear encountered during processing. Also, Brookfield viscometers measure apparent viscosity at constant temperatures and shear rates, and this limits their ability to capture the dynamic thermal transitions associated with starch gelatinization (Nastasi et al. 2025). On the other hand, the RVA provides a small-sample, standardized, and time-efficient method that closely mimics industrial operations like extrusion, cooking, and formulation (Tian *et al.* 2022). Thus, the RVA has become an essential tool for evaluating the functional performance of native and modified starches, most especially in relation to their processing property and end-use applications.

Notwithstanding wide investigations into starch modification, especially *via* acid hydrolysis and oxidation processes, vital gaps remain in the comprehensive understanding of how oxidation conditions influence the interrelationship between pasting behavior, structural transformation, and application-relevant functional properties of maize starch. Preceding investigations have largely focused on isolated aspects of starch modification including structural characterization or selected functional features, without systematically integrating them to establish clear structure–property–function relationships. Also, other findings emphasize generalized acid modifications without detailed consideration of controlled alkaline oxidation systems and their specific mechanistic implications on starch functionality. In addition, limited interest has been given to the combined assessment of Rapid Visco Analyzer (RVA) pasting profiles alongside morphological and molecular analyses, under well-defined oxidation conditions. This limitation restricts the ability to fully explain how oxidative modifications at the molecular level translate into functional performance during mechanical and thermal processing. The current work aimed to address these limitations by employing a controlled alkaline sodium hypochlorite (NaOCl) oxidation system (pH 9 to 9.5) to modify maize starch, followed by a comprehensive and systematic characterization of its physicochemical, structural, and functional properties.

This work thus focused on: (i) the use of precisely controlled oxidation conditions to effect targeted structural changes; (ii) the integrated analysis of RVA pasting behavior with SEM/EDX, FTIR, and functional property measurements; and (iii) the establishment of mechanistic correlations between oxidation-induced molecular modifications and macroscopic functional performance. By linking structural transformations such as granule disruption and carbonyl group formation with observed changes in gelation behavior, swelling capacity, viscosity profiles, and water/oil absorption capacities, this work provides deeper mechanistic insight into the behavior of oxidized maize starch.

MATERIALS AND METHODS

Sourcing for Maize and Extraction of Starch

Raw maize (*Zea mays* L.) in this study, was obtained from Mowe market in Ogun state, Nigeria. The maize was washed, crushed, and stored at room temperature in airtight containers until further use to prevent moisture uptake and contamination. Starch was extracted from maize through a process called wet milling. The maize was soaked in water for 3 days to soften them. The soaked maize was then washed with distilled water and ground into a coarse slurry using a mechanical grinder. The germ was removed through centrifugation at 3500 rpm. The mixture was left for 12 h to settled and the starch

which latter condensed at the bottom of the container was obtained *via* decantation of the water layer and washed again with distilled water. The purified starch was then dried in an oven at 80 ºC for 3 h, and the resulting fine powdery substance was referred to as raw maize starch (RMS).

Activation of Starch

The employed modification process in this study corresponds to oxidation with sodium hypochlorite as the solvent under controlled alkaline conditions, rather than acid hydrolysis. The alkaline medium (pH 9 to 9.5) enabled the formation of carboxyl and carbonyl groups, resulting in functional and structural modifications of the starch.

Oxidation of the starch was performed by adopting the method described by Lawal *et al.* (2005) with little adjustments. 15.8% of starch slurry was prepared by dissolving 100 g of starch in 500 mL of distilled water followed by pH was adjustment to 9.5 using 2 M NaOH. Thereafter, 10 g of NaOCl was added and stirred for 30 min, while keeping the pH within range of 9-9.5 with constant stirring. Thereafter, the pH was further adjusted to 7 using 1 M H_2SO_4, and the oxidized starch formed was filtered, washed with distilled water and air-dried. The product obtained was referred to as oxidized maize starch (OMS). In this study, the raw maize starch sample was used as the control sample, while oxidized maize starch represented the modified system. The study focuses on the evaluation of the influence of oxidation conditions relative to the native starch baseline.

Characterizations Maize Starch

Gelatinization studies

The method of Lin *et al*. (2017) was used to investigate gelation property of the starch. Samples of starch, 2% to 18%(w/v), were made in test tube with distilled water (10 mL). The starch suspensions were stirred for 5 min and heated at 80 ºC for 30 min in a water bath, followed by rapid cooling under running cold water tap. The contents were further cooled for 2 h at 4 ºC.

Oil and water absorption capacity

The approach described by Lawal *et al.* (2005) and Jung *et al*. (2017) was used to estimate the water and oil absorption capacities of the starch. About 1 g of sample was dissolved in 10 mL of distilled water or oil and the content was thoroughly stirred and allowed to stand for 30 min. Then, the water absorption capacity (WAC) as well as the oil absorption capacity (OAC) were estimated using Eqs. 1 and 2 below,

$$WAC = {}^{RW}/_{WS} \quad (1)$$

$$OAC = {}^{RW}/_{WS} \quad (2)$$

where RW denotes the residue weight of starch (g) and WS is the weight of the starch (g).

Pasting Properties

The pasting characteristics of starch were found using the Rapid Visco Analyzer (RVA; RVA-4, Newport scientific, Australia). About 2.5 g dried starch sample was dissolved in 25 mL of distilled water and the sample was heated for 30 min from 50 ºC to 95 ºC, held at 95 ºC for 2 min and cooled to 50 ºC within 3 min. The gelatinization

parameters like peak viscosity, maximum gelatinization temperature, holding strength, final viscosity, breakdown and set back were then measured.

Fourier Transform Infra-Red Spectroscopy

Fourier transform infra-red (FT-IR) spectroscopy investigates the various functional groups present in starch powder before and after treatment were carried out using TENSOR 27 series FT-IF spectrometer, Germany. The KBr pellet technique was used in which 1.99% of the powder and KBr is combined in a mortar and pestle and then compressed to a 2 mm diameter pellet ratio. All FT-IR spectra data were recorded in the range of 4000 cm^{-1} to 400 cm^{-1} with resolution of 4 cm^{-1} and 64 times scanning.

Scanning Electron Microscopy

The morphology and chemical analysis of the starch granules was examined using a scanning electron microscopy (SEM) which was coupled with energy dispersive X-ray (EDX) (Tescan Vega 3, Czech Republic). All of the samples were cooled with gold and examined in the scanning electron micro-scope under an acceleration.

Solubility Swelling Power Determination

Effects of temperature on solubility and swelling power were evaluated in the temperature range of 55 to 95 ºC by adopting the method described by Dhull *et al.* (2021). Approximately 0.1 g of starch sample was added to a clean dried test tube and weighed (W_1). Then 10 mL of distilled water was added, and the content was thoroughly mixed. The slurry formed was heated within the temperature range of 55 to 95 ºC for 30 min inside water bath. The content was cooled to room temperature and centrifuged for 15 min at 4500 rpm. The product formed after centrifugation with the water was retained and the test tube was weighed (W_2). The swelling power (SP) was estimated using equation 3 below,

$$SP = \frac{W_2 - W_1}{WS} \times 100 \quad (3)$$

where WS is the weight of the starch in g and W_1 and W_2 are as previously defined.

Statistical Analysis

All experiments were performed in triplicate (n = 3), and the values are presented as mean ± standard deviation. Statistical analysis was performed using one-way analysis of variance (ANOVA) to determine significant differences between raw maize starch (RMS) and oxidized maize starch (OMS). Differences were considered statistically significant at $p < 0.05$.

RESULTS AND DISCUSSION

Gelatinization Analysis

The gelation characteristics of RMS and OMS samples at diverse concentrations are presented in Table 1. At 2% (w/v), both OMS and RMS remained entirely liquid and showed no sign of gel formation, signifying that the polymer content at this concentration was inadequate to provide network development in either starch. But when the

concentration was increased to 5%, their properties diverged markedly. OMS produced a cohesive and firm gel, whereas, RMS formed only a viscous and weak gel.

This improved gelation capacity is consistent with structural changes documented for acid-hydrolyzed starches, where interruption of crystalline regions and partial cleavage of amylose and amylopectin chains increased swelling and polymer release during heating (Li and Hu 2021; Boonkor *et al*. 2022). Acid hydrolysis is also known to breakdown the granular matrix, allowing easier gelatinization as well promoting closer intermolecular packing during cooling (Gong *et al*. 2024).

At 10% (w/v), OMS yielded a very firm gel, while RMS reached its least gelation concentration, forming a stable but fairly soft gel. Stronger gels in modified systems have been ascribed to condensed polymer chains and alterations of the granular ultrastructure, which enable tighter junction-zone formation and denser gel networks (Kibar *et al*. 2024; Mao *et al*. 2024). Overall, the concentration-dependent property clearly proves that the modification meaningfully improves the gelation characteristics of maize starch, permitting stronger gels to form at lower concentrations which is an observation well reinforced by recent studies on physically and chemically modified starches (Gong *et al*. 2024).

Table 1. Gelatinization Analysis of OMS and RMS

Sample	Concentration (% w/v)	Gel Formation (Yes/No)	Gel Strength Description	Gel Strength Score*
RMS	2	No	Liquid	0
RMS	5	Partial	Weak/viscous gel	1
RMS	10	Yes	Soft gel	2
OMS	2	No	Liquid	0
OMS	5	Yes	Firm gel	3
OMS	10	Yes	Very firm gel	4

* **Notes:** Gel strength score is a semi-quantitative scale based on visual inversion and structural stability: 0 = no gel (liquid),1 = weak/flowing gel, 2 = soft gel (partially self-supporting), 3 = firm gel (self-supporting), 4 = very firm gel (rigid, non-flowing)

Oil and Water Absorption Capacity (WAC) Analysis

Table 2 presents the water and oil absorption capacities (WAC and OAC) of RMS and OMS. These parameters describe how starch interacts with polar and non-polar substances, which influences its functional performance in food and industrial systems. The WAC value of OMS (1.5±0.01 g/g) was found to be higher than that of RMS (0.9±0.01 g/g), indicating that the starch's ability to bind water was enhanced by the modification. The ordered crystalline regions of starch granules are partially disrupted by the oxidation process. As a result, additional hydroxyl groups are exposed and they can form hydrogen bonds with water molecules (Zarski *et al*. 2024; Xie *et al*. 2025). The rise in WAC suggests greater porosity and hydrophilicity, corroborating literature reports that oxidized starches exhibit improved swelling and hydration property because of structural loosening (Gong *et al*. 2024). Likewise, the modification also improved the starch's interaction with hydrophobic compounds; this was evidenced by the rise in the OAC from 0.6 g/g in RMS to 1.2 g/g in OMS. This could be as a result of the surface exposure and partial degradation of non-polar sites, which enables more oil entrapment (El Farkhani *et al.* 2024; Gong *et al*. 2024).

Table 2. Water and Oil Absorption Capacities of RMS and OMS

Parameters	RMS (g/g)	OMS (g/g)
Water Absorption capacity (WAC)	0.9±0.01	1.5±0.01
Oil Absorption Capacity (OAC)	0.6±0.01	1.2±0.02

Values are expressed as mean ± standard deviation (n = 3).

Pasting and Viscosity Properties Results

The results from RVA of OMS and RMS samples are represented in Figs. 1 and 2, respectively and the parameters values depicted in Table 3. The pasting and viscosity properties of starches provide essential perception into their functional performance during processing operations such as shearing, heating, and cooling. The data reveal considerable differences in the rheological characteristics of maize starch following modification. The peak viscosity, which represents the maximum swelling capacity of starch granules in the gelatinization process, was slightly reduced in OMS (1987 cP) compared to RMS (2089 cP). This reduction suggests that oxidation partly degraded the amylose and amylopectin molecules, weakening the internal granule structure and constraining its swelling potential (Halim *et al*. 2024).

The trough viscosity also declined slightly from 1916 cP in RMS to 1879 cP in OMS, demonstrating that the treatment reduced the paste's ability to sustain viscosity under continuous heating and shear (Xing *et al*. 2017; Compart *et al.* 2023). The breakdown viscosity (difference between trough and peak) was particularly lower for OMS (108 cP) compared to RMS (173 cP), implying that the modified starch paste had greater shear and thermal stability. The final viscosity, which specifies the ability of starch molecules to reassociate in cooling process, decreased noticeably from 2554 cP in RMS to 2110 cP in OMS. Similarly, the setback viscosity (final minus trough viscosity) declined from 638 cP in RMS to 231 cP in OMS. Lower final and setback viscosities reflect a weakened tendency for retrogradation (the process of recrystallization of gelatinized starch molecules in cooling). Declined retrogradation is beneficial in products requiring low syneresis and paste stability, such as sauces, frozen desserts, and fillings (Boonkor *et al.* 2022). A study by Liu *et al*. (2024) on rice starch with added proteins (SPI / WPI) reported that extrusion plus protein addition caused a delayed retrogradation and reduction in the setback (SB) viscosity compared with native rice starch which improves the stability of starch-based foods. Similarly, Qiu *et al*. (2024) in their review observed that extrusion often decreases breakdown, peak, final, and setback viscosities, which matches with reduced retrogradation tendency

The pasting temperature of OMS (348.7 K; ≈75.7 °C) was partly lower than that of RMS (351.0 K; ≈78 °C), signifying that oxidation weakens the granular matrix, allowing gelatinization to take place at lower temperatures. This discovery is consistent with earlier studies reporting that modified starches gelatinize more readily because of partial disruption of crystalline regions. The peak time also rose marginally from 352.2 s to 376.2 s, probably indicating slower hydration and rearrangement of starch molecules during pasting. Puspitasari *et al.* (2025) examined the effects of citric acid concentration and hydrolysis time on starch and observed that partial acid hydrolysis lowers gelatinization temperature, cuts amylopectin/amylose chains, and changes pasting behaviour.

Compared with RMS, the OMS sample demonstrated lower peak, trough, and final viscosities, and a markedly declined setback, consistent with partial hydrolysis of amylopectin and amylose which restricts granule swelling and reduces retrogradation tendency (Halim *et al*. 2024). The decrease in the breakdown of the OMS further suggests improved paste stability under heat and shear, a property commonly reported for modified-thinned starches where molecular depolymerization yields smaller, less rupture-prone fragments (Xing *et al*. 2017; Compart *et al.* 2023). The slight change in peak time and small decrease in pasting temperature are also consistent with altered hydration dynamics and weakened granular crystallinity following treatment (Thakur *et al*. 2021). Collectively, the observed changes indicate that modification shifted maize starch from a high-viscosity, gel-forming material toward a lower-viscosity, more process-stable ingredient suited to applications requiring smooth, stable pastes and reduced syneresis (*e.g.*, sauces, fillings, confectionery), while native RMS would remain preferable where strong thickening and gel strength are desired (Karma *et al*. 2022; Compart *et al.* 2023).

Table 3. Pasting and Viscosity Properties of RMS and OMS as Measured by RVA

Sample Parameters	RMS	OMS
Pasting temperature (K)	351	348.7
Peak viscosity	2089	1987
Final viscosity	2554	2110
Peak time (S)	352.2	376.2
Setback viscosity	638	231
Breakdown	173	108
Trough viscosity	1916	1879

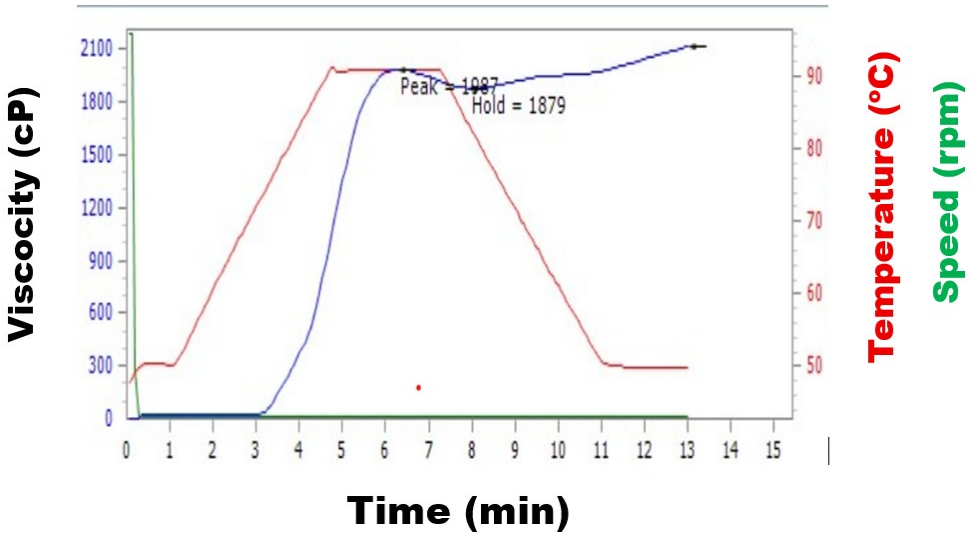

Fig. 1. Graph of viscosity against time for OMS

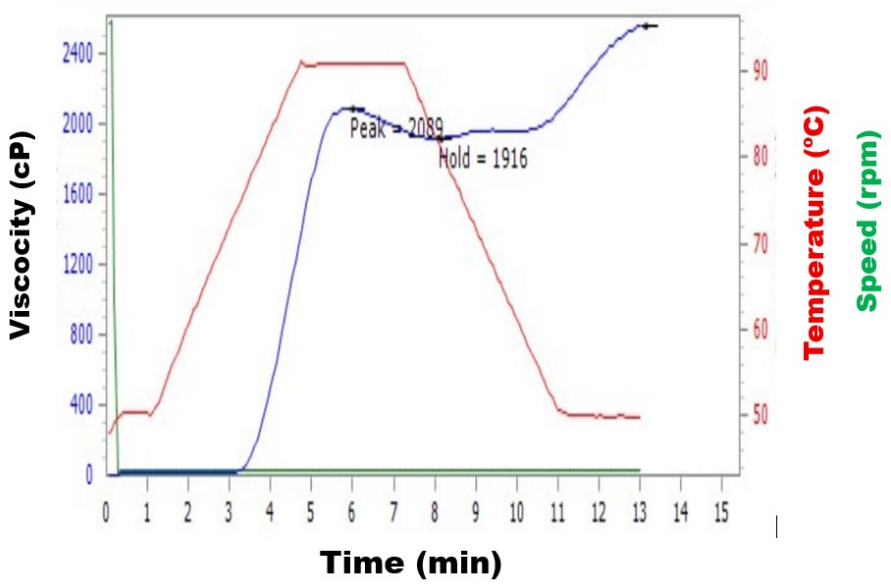

Fig. 2. Graph of viscosity against time for RMS

Swelling Power

The swelling characteristics of RMS and OMS across temperatures is represented in Table 4. It was observed that at all temperatures examined (55 to 95 °C), OMS displayed considerably greater swelling power compared to RMS, signifying that modification significantly transformed the granular integrity and hydration capacity of the starch. Even at a sub-gelatinization temperature of 55 °C, OMS (0.43±0.12 g/g) already exhibited much superior swelling than RMS (0.26±0.24g/g), signifying that the treatment had weakened the granular matrix and made the granules more available to water at low thermal input. As the temperature rose to 65 and 75 °C, both starches swelled more, but the variation between them enlarged. RMS improved modestly from 0.81±0.32 to 1.03±0.13 g/g; OMS surged from 1.04±0.22 to 1.68±0.04 g/g, indicating greater sensitivity of the modified granules to thermal disruption. At higher temperatures of 85–95 °C, the variation became even more noticeable: RMS attained 1.61±0.21 g/g, whereas, OMS swelled to 2.78±0.13 g/g. These improved swelling levels for OMS are consistent with the general principle that chemical modification of starch can break down amorphous and crystalline regions, reduce amylose/amylopectin chains, and thus improves swelling capacity and water accessibility (Li and Hu 2021; Zhang *et al*. 2023b).

The improved swelling power of OMS also could be as a result of increased hydrophilicity and exposure of hydroxyl groups after hydrolysis, which inspires water uptake (Zhang *et al*. 2023b; Konował *et al*. 2024). In contrast to some reports of modified starches indicating reduced solubility or swelling (depending on concentration, treatment conditions, and solvent type) (Arachchi *et al*. 2025), data from this study demonstrated that under the present modification conditions, the oxidation process caused an increased swelling across the full temperature range. This indicates that the solvent type, degree of hydrolysis, and experimental conditions seriously affect swelling behavior, and that under mild-to-moderate hydrolysis, maize starch can gain enhanced expansion capacity and water-binding affinity.

Table 4. Swelling Power of OMS and RMS at Various Temperatures

Temperature/ Samples	55 ºC	65 ºC	75 ºC	85 ºC	95 ºC
SP for RMS (g/g)	0.26±0.24	0.81±0.32	1.03±0.13	1.44±0.23	1.61±0.21
SP for OMS g/g)	0.43±0.12	1.04±0.22	1.68±0.04	2.05±0.11	2.78±0.13

Values are expressed as mean ± standard deviation (n = 3).

FT-IR Results

FT-IR was used to identify the functional groups of the components in the RMS and OMS, as shown in Fig. 3. The FT-IR spectra of the raw maize starch demonstrated the vibrational characteristics associated with native starch polysaccharides exemplified by a broad absorbance band around 3357 to 3486 cm^{-1} that can be attributed to O–H stretching of intermolecular hydrogen bonds (Subroto *et al*. 2023; Khumalo *et al*. 2024). The peak seen at 2924 cm^{-1} arises from C–H stretching of the glucopyranose rings, while the peak seen at 1745 cm^{-1} can be assigned to C=O stretching (Subroto *et al*. 2023; Khumalo *et al*. 2024).

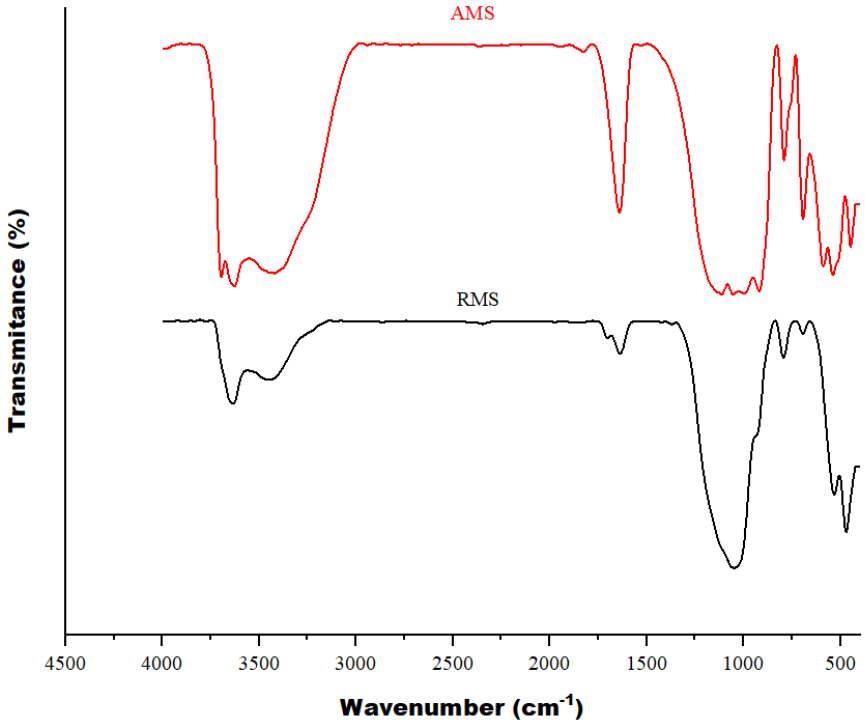

Fig. 3. FT-IR of OMS and RMS samples

The peaks between 1152 to 950 cm^{-1} gave strong C–O, and C–O–C stretching vibrations characteristic of glycosidic linkages in starch (Subroto *et al*. 2023; Khumalo *et al*. 2024). Upon the oxidation of the maize starch (OMS), the fundamental bands corresponding to polysaccharide still remained present, affirming the preservation of the starch backbone after treatment. Nevertheless, certain notable differences were seen suggesting successful modification. OMS exhibited increased intensity with slight band shift with the O–H stretching, signifying altered hydrogen-bonding patterns because of oxidation of hydroxyl groups. More importantly, an enhanced absorption band was seen at 1753 cm^{-1} region, which can be assigned to the C=O stretching. Additional

modifications were observed in the regions spanning across 1252 to 1035 cm^{-1}, corresponding to C–O–C, C–O, and C–O–C stretching vibrations.

SEM and EDX Analyses/Results

SEM micrographs of the grains of raw and oxidized starch samples are presented in Fig. 4. The RMS exhibited morphology characteristics of native starch, consisting of polygonal granules or spherical particles, and intact surfaces. These granules seemed uniformly dispersed and showed no signs of structural disruption, indicating crystalline–amorphous organization typical of untreated starch. On the contrary, the OMS demonstrated a remarkedly altered morphology with lost native granular and irregular structures. The new morphology was in the form of sheet-like fragments, which are indicative of possible extensive hydrolytic degradation. The surfaces appeared eroded, rough, and consistent with the preferential attack on the amorphous regions, causing progressive granule breakdown, pore formation, and reduced structural integrity (Liu *et al*. 2021). Also, the lack of grain-like features in the SEM of the OMS is consistent with the gelatinization of the starch, essentially converting it to a water-soluble form.

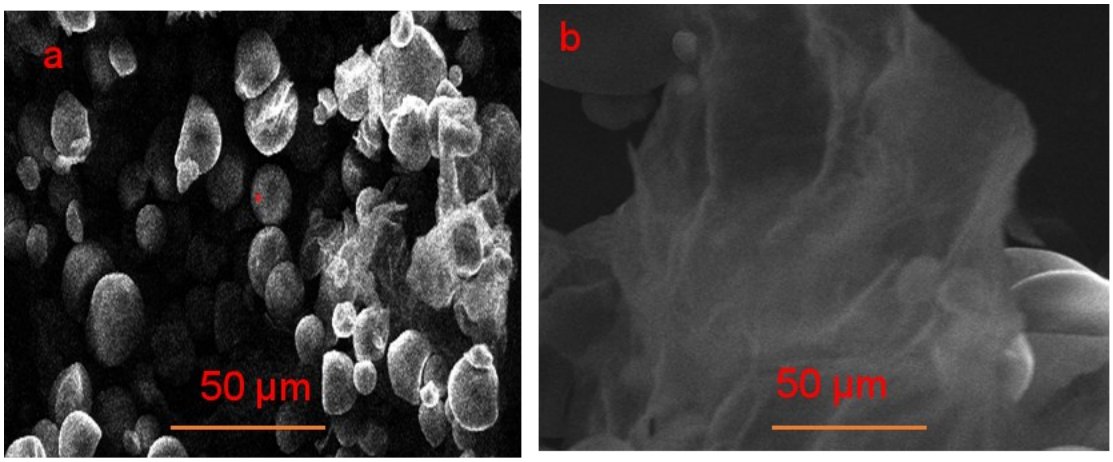

Fig. 4. SEM of (a) RMS and (b) OMS samples

The chemical composition characteristics of the starch samples before and after treatment are as listed in Fig. 5. The EDX spectra of the RMS (Fig. 5a) reveal that it's primarily composed of carbon (C) and oxygen (O), which is typical of polysaccharide-based biopolymers. The dominant carbon and oxygen peak is an indication that the compound composed of carbohydrate structure of starch (–C–C–, –C–O–, and –C–O–C– linkages), while the absence of other essential elemental is an indication of the purity of the starch granules. Following oxidation, the EDX spectrum of OMS (Fig. 5b) retains the same elemental profile, with carbon and oxygen as the only detectable elements. However, a noticeable rise in the relative intensity in the peak corresponding to oxygen was observed when compared to carbon. This shift indicates increased exposure or partial oxidation of carbonyl and hydroxyl groups as a result of the treatment. The treatment most likely attacks amorphous regions of starch, breaking glycosidic bonds and generating more oxygen-rich functional groups.

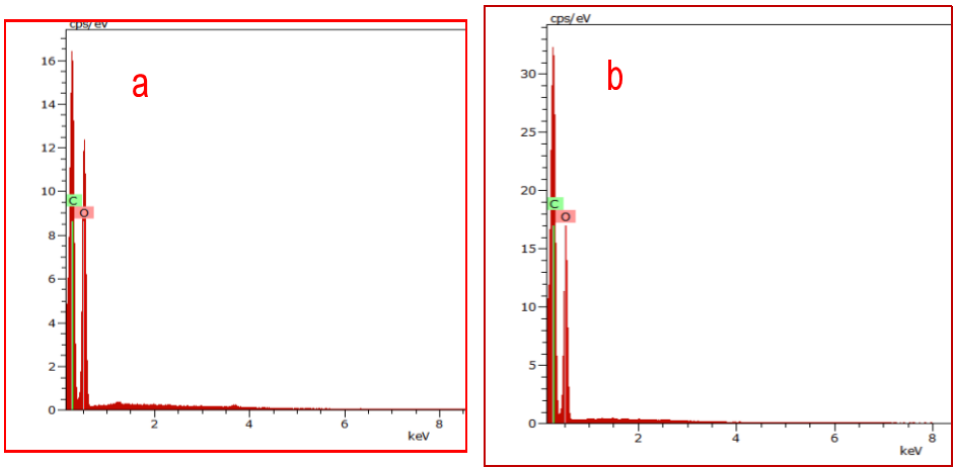

Fig. 5. EDX of (a) RMS and (b) OMS samples

Comparative Evaluation with Previous Studies

The modification-induced changes observed in this study are generally consistent with prior reports on chemically treated starches; however, several important distinctions emerge when compared critically with existing literature. For instance, the reduction in peak, final, and setback viscosities of the oxidized maize starch aligns with findings from acid-hydrolyzed and oxidized starch systems, where molecular depolymerization reduces granule integrity and limits swelling capacity. Acid hydrolysis has been reported to decrease starch molecular weight and disrupt amorphous regions, thereby causing significant reductions in peak and final viscosities due to diminished swelling and paste development (Javadian *et al*. 2021). In addition, oxidation processes facilitate chain cleavage and the introduction of carboxyl and carbonyl groups, causing reduction in viscosity parameters such as peak and final viscosity, as well as decreased setback values linked with suppressed retrogradation (Liewchirakorn and Ngamchuea 2023).

Nevertheless, the magnitude of reduction in setback viscosity observed in this study is comparatively greater than that reported in some acid-modified maize starch systems, suggesting a more pronounced suppression of retrogradation under the present oxidation conditions. This feature may be attributed to enhanced electrostatic repulsion and greater disruption of starch chain interactions following oxidative modification.

Interestingly, in contrast to studies reporting decreased swelling power following extensive acid hydrolysis due to excessive chain scission and loss of granular integrity, the oxidized starch in this work exhibited consistently higher swelling across the temperature range studied. There has been report of reduced swelling power as a result of molecular degradation and the formation of shorter dextrin chains that weaken the granule structure and reduce its water-holding capacity because of acid hydrolysis (Kaur *et al*. 2011). On the other hand, oxidation treatments may exert dual influences depending on the severity of the reaction. While severe oxidation can lead to reduce swelling because of structural disintegration, moderate oxidation introduces carboxyl and carbonyl groups that facilitate hydrophilicity and enhanced water absorption (Sumardiono *et al*. 2021).

The controlled alkaline NaOCl system employed here likely induced partial oxidation and disruption of amorphous regions without extensive degradation of the crystalline framework, thereby enhancing water accessibility while preserving sufficient structural integrity for granule expansion. This suggests that swelling behavior is not

solely dependent on modification type but is highly sensitive to the degree of molecular alteration, an aspect that remains insufficiently addressed in many previous studies. Similar findings have been documented in oxidized starch systems, where low to moderate oxidation levels facilitate hydration, while more severe treatments (either acid-based or oxidative) result to reduced swelling because of granule disruption (Vanier *et al*. 2012).

Table 5. Comparative Analysis of Viscosity Reduction and Swelling Trends in Modified Starches

Modification Type & Conditions	Change in Viscosity	Swelling Behavior	Mechanism	References
Alkaline NaOCl oxidation (pH 9 to 9.5)	Peak viscosity ~4.9%; Setback ~63.8%	Swelling ~70 to 75% (at 95 °C)	Partial depolymerization and increased hydrophilicity	Present study (OMS)
Oxidation & acid thinning (maize starch)	Decline in peak, hot paste, and setback viscosity	Swelling compared to native	Weakening of structural but increased solubility; oxidation reduces swelling	Lawal *et al*. (2005)
Sulfuric acid hydrolysis (waxy maize)	Viscosity alteration because of molecular size reduction	Reduced or altered swelling depending on chain degradation	Depolymerization of amylopectin chains controls rheology	Li *et al.* (2020)
Acid hydrolysis + heat-moisture treatment	Reduction in viscosity parameters depending on temperature and pH	Swelling significantly at ≥75 °C	Increased crystalline stability and amylose–lipid interactions limit swelling	Sun *et al.* (2015)
Oxidized starch (legume system)	Functional weakening linked with modification	Swelling (~5–10% reduction)	Reduction in granule expansion due to structural disintegration	Guleria and Yadav (2022)
Acid / dual modification (cereal starch)	Declined viscosity with hydrolysis severity	Swelling (initially), then with prolonged treatment	swelling increases with mild hydrolysis; severe hydrolysis collapses structure	Biduski *et al.* (2017)
Acid-modified maize starch (HCl, organic acids)	Increased solubility and reduced viscosity	Increased swelling (acid-dependent)	Reduced water-binding capacity due to acid attack on amorphous regions	Skendi *et al.* (2020)

The FTIR results further support this interpretation, where the increased intensity of carbonyl-related bands indicated successful oxidation of hydroxyl groups. This chemical transformation can be directly linked to the observed increase in water absorption capacity, as the introduction of polar functional groups enhances hydrogen bonding with water molecules. Similarly, SEM observations of surface erosion and loss of granular integrity provide morphological evidence for the enhanced swelling and gelation behavior. These findings collectively demonstrate a clear structure–property relationship, where oxidation-induced molecular and microstructural changes govern

macroscopic functional performance. Despite these insights, discrepancies across studies highlight existing knowledge gaps. For example, while both acid hydrolysis and oxidation are known to reduce viscosity, their relative effects under comparable conditions remain poorly quantified, limiting the ability to predict functional outcomes. Additionally, the long-term stability and retrogradation kinetics of oxidized starches under storage conditions are still not well understood. Addressing these gaps will require more systematic studies that directly compare modification techniques under controlled and standardized conditions.

Limitations and Future Work

While this current study has provided detailed insight into the influence of controlled oxidation on maize starch properties, it did not systematically examine the effect of key reaction parameters including reaction time, oxidant concentration, and pH variation. These factors are known to significantly influence the degree of oxidation and resulting functional properties. Although this work gave insight into oxidation-induced modifications, it did not include comparative treatments such as acid hydrolysis or varying oxidation intensities. Thus, future works should focus on the optimization of these conditions to establish more precise structure–property relationships and enhance application-specific performance. In addition, inclusion of such conditions in future works to establish more comprehensive structure–property relationships.

CONCLUSIONS

1. The oxidation of maize starch introduced some features into the functional, physicochemical, and structural properties when compared to its native form.
2. The Rapid Visco Analyzer (RVA) results revealed reduced peak, final, and setback viscosities, suggesting lower retrogradation and better shear and thermal stabilities.
3. The Fourier transform infrared (FT-IR) investigation confirmed increased intensity with slight band shift in functional groups such O–H, C=O, and C–O–C stretching after treatment, while scanning electron microscope (SEM) and energy dispersive X-ray (EDX) showed alterations in granule structure and elemental composition after the oxidation process.
4. Oxidized starch showed higher oil and water absorptions, increased swelling at all temperatures, and stronger gel formation at lower concentrations.
5. These findings indicate potential functional advantages in applications requiring enhanced hydration and reduced retrogradation; but, direct validation in real food systems was not performed and remains an important area for future investigation.

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