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# Impact of Acetylation and Oxidation on Some Functional, Structural and Pasting Properties of Irish Potato (*Solanum tuberosum*) starch

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**ABSTRACT**

**To meet the demanding technological needs of today, as well as eliminate the drawbacks, native starches have been chemically modified. Oxidation and acetylation have been widely employed to alter the chemical structure of starch. In this study, native Irish potato starch (NIPS) was chemically modified with acetic anhydride and sodium hypochlorite to produce oxidized (OIPS) and acetylated (AIPS) starch. The functional and pasting properties of NIPS, OIPS and AIPS were investigated. Oxidation and acetylation decreased the bulk density, pH, and water absorption capacity (WAC) of native Irish potato starch whereas an improvement was observed in the moisture content, the oil absorption capacity (OAC) and foam capacity with respect to chemical modification. Oxidation and acetylation increased the OAC of NIPS by 26.7% and 138%, and the foam capacity by 23.1% and 34.0%. Oxidation and acetylation decreased WAC by 18.75% and 61.27% respectively. Oxidation increased the dispersibility by 1.47% while acetylation decreased it by 4.94%. Oxidation increased peak and trough viscosities but reduced breakdown, final and setback viscosities, whereas acetylation increased peak, trough and breakdown viscosities but reduced final and setback viscosities. The high peak and final viscosities of NIPS, OIPS, and AIPS suggest that they could be used as thickeners in sterilizable products such as baby food and sauces.**

## 1. INTRODUCTION

Starch, commonly known as amylum, is a type of polysaccharide carbohydrate made up of several glucose units connected by glycosidic bonds. It is the primary carbohydrate reserve in grains, tubers, and legumes (Olatunde *et al*., 2017). It consists of discrete semi-crystalline granules with 20–30% linearly organized amylose and 70–80% highly branched amylopectin chains. (Bajaj *et al*., 2018). Starch granules differ in structural and chemical properties, as well as how amylose and amylopectin are distributed throughout the structure, depending on their botanical origin (Wang & Copeland, 2015; Olatunde *et al*., 2017). The organization of the starch granules and the amylose/amylopectin ratio determine the starch's functional qualities, which in

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turn dictate the starch's vast array of industrial uses in foods, medicines, and adhesives (Tharanathan, 2005; Olatunde *et al*., 2017). Because of its low shear stress endurance and heat disintegration, coupled with significant retrogradation and syneresis, starch in its native state has limited industrial applications, albeit these weaknesses can be solved by starch modification (Colussi *et al*., 2014; Halal *et al*., 2015; Yussuf *et al*., 2022). In order to improve their uses both in the food and non-food sectors, native starch granules are typically modified to improve their advantageous attributes and get rid of their drawbacks, such as low pH, facile retrogradation, decreased viscosity, gelling, and syneresis after cooking (Suri & Singh, 2023). Diverse methods have been employed to modify starch for diverse reasons in industrial applications, which can be broadly classified into physical, chemical, and enzymatic modifications (Colussi *et al*., 2014; Halal *et al*., 2015; Subroto *et al*., 2023). Chemical modification of starches through oxidation, acetylation, esterification, etherification, acid thinning, crosslinking, succinylation and phosphorylation have been employed to increase its intrinsic qualities, such as digestion, thickening power, solubility, pasting properties, and shear stability(García *et al*., 2012; Kalita *et al*., 2014; Isah *et al*., 2015; Isah, 2018; Ibikunle *et al*., 2019, Oderinde *et al*., 2020; Ramadan & Sitohy, 2020; Zięba *et al*., 2021; Ibikunle *et al*., 2022; Oderinde *et al*., 2023). This process includes the blocking or introduction of a functional group to the starch molecules to change the bulk properties (Liu *et al*., 2022; Suri & Singh, 2023). Starch acetylation is a chemical alteration in which a portion of the glucose monomers' hydroxyl groups are changed into acetyl groups, hence modifying the starch's molecular structure (Halal *et al*., 2015). Acetylation is a common chemical modification method and it is produced by the reaction of native starch with reactive agents such as acetic anhydride, octenyl succinic anhydride (OSA), or vinyl acetate with an alkaline catalyst presence (KOH, NaOH, $Na_2CO_3$, $Ca(OH)_2$) (Alcázar-Alay & Meireles, 2015; Halal *et al*., 2015). Native starch loses its initially organized structure when acetyl groups are added, and the electrostatic interaction between the chains of amylose and amylopectin is also diminished. Several factors influence starch acetylation, including the kind of starch, the concentration of the reactant, the type of catalyst, the reaction duration, and the pH of the suspension (Halal *et al*., 2015). Acetylated starches are used in both food and non-food sectors for salad dressings, filled cakes, gravies, fruit pies, gummed tape adhesives, biodegradable films, surface-sizing for papers, wrap-sizing for textiles, thickeners, encapsulating agents, adherents, stabilizers (Alcázar-Alay & Meireles, 2015; Halal *et al*., 2015). Acetylation has been shown in earlier research to enhance the solubility and water absorption of starch while delaying starch retrogradation (Liu *et al*., 2022).

Starch oxidation involves the addition of an oxidizing agent to a native starch at a regulated temperature, pH and reaction time thereby introducing a carboxy and carbonyl groups into the starch molecules. At first the hydroxyl group are oxidized to a carbonyl group and then to a carboxyl group (Halal *et al*., 2015; Suri & Singh, 2023). Several oxidants, including, peracetic acid, nitrogen dioxide, hydrogen peroxide, potassium permanganate, ozone, sodium hypochlorite, and chromic acid, can

be used to oxidize starch. The most commonly used is sodium hypochlorite and ozone. The degree to which starch is oxidized by hypochlorite is determined by a number of variables, including the molecular structure of the starch, its origin, the size of amorphous lamellae and the packing of crystalline lamellae, temperature, the pH of the solution, reaction time, and the concentration of oxidants and catalyst. Starch oxidation frequently raises the retrograded starch's melting point while decreasing its viscosity during pasting, the swelling power, and enthalpy change during gelatinization. Oxidized starches find application in the textile, paper, and laundry sectors, as well as in batters, breading, food coating, confectionery, and dairy as binders and texturizers (Halal *et al*., 2015; Suri & Singh, 2023).

The Irish Potato (*Solanum tuberosum L.*) is a starchy, tuberous crop that is often referred to as "white potato." It belongs to the Perennial Solanum Tuberosum family of the Solanaceae family, which is often known as "the nightshades". The Irish potato ranks fourth in terms of overall crop production, just after rice, wheat, and corn, and is regarded as the most significant non-cereal crop in the world. In general, potatoes are high in starches and glycoalkaloids (Padmanabhan *et al*., 2015). Despite being rich in starch content, only the physicochemical properties of sweet potato starch have been extensively explored. There is no published study on the properties of oxidized and acetylated Irish potato starch. The objective of this research is to investigate the effects of oxidation and acetylation on some functional and pasting properties of Irish potato starch.

## 2. MATERIALS AND METHODS

### *2.1. Starch Extraction*

Tubers of Irish Potato (*Solanum tuberosum L.*) were acquired from a local market in Ijebu Ode, Ogun State, Nigeria. The starch isolation was done using the method described by Nwokocha *et al*., (2014). Tubers of Irish Potato were peeled and washed with tap water. The peeled Irish potato tubers were chopped into small pieces, thoroughly washed with distilled water and grounded into slurry with a blender. Distilled water was added to the slurry during the sieving in order to allow easy passage through the filter. The filtrate was allowed to settle for 4 hours after which the starch was isolated and the supernatant was decanted. The washing was done for additional 4 times. The extracted starch was exposed to sunlight to get rid of the moisture.

### *2.2. Preparation of Acetylated Starch*

The acetylation of starch was achieved by method described in 2004 Lawal, (2004). 100 g of NIPS is dispersed in 500 ml of distilled water and stirred for 40 min. The pH is adjusted to 8.0 with 1.0 mol of NaOH, and the reaction is run for 10 min. The pH of the slurry was kept between 8.0 and 8.5 by the addition of 10.2 g of acetic anhydride. Finally, the pH of the mixture was reduced to 4.5 with 0.5 mol of HCl. The mixture is then filtered, the residue of the filtrated sample is then washed thrice with distilled water and dry at room temperature for about 48 hours.

### ***2.3. Preparation of Oxidized Starch***

Oxidation of the starch was done by method described in 2004 by Lawal, (2004). 100 g of NIPS is dispersed in 500 ml of distilled water and stirred for 30 min, and then the pH of the slurry was raised to 9.5 using 4.0

M of NaOH. 10 g of NaOCl was added to the slurry within a period of 30 min, with constant stirring while maintaining a pH between 9.0 – 9.5. After adding NaOCl, the reaction was allowed to continue for 10 min. The pH was then adjusted to 7.0 using a few drops of a 1 M solution of $H_2SO_4$. Subsequently, the oxidized starch was filtered and rinsed four times with distilled water and air-dried at about 30±2 °C for 48 hrs.

*2.4. Determination of Moisture Content*

5 g of native and modified starch samples were weighed in an automatic moisture analyser, and the temperature was set to 105 °C for 30 min or more. The moisture percentage of the starch was then displayed on the screen of the analyzer.

$$Moisture\ content = \frac{Average\ change\ in\ volume}{Initial\ volume} \times 100$$

(1)

*2.5. Physicochemical Properties*

*Determination of pH*

Native and modified starch samples (10 g) were weighed in triplicate into a beaker and mixed with 40 ml distilled water. The resultant suspension was agitated for 5 min before allowing to settle for 10 min. The pH of the supernatant was determined using a calibrated pH meter ( Yussuf *et al.,* 2018)**.**

*Determination of Bulk Density*

The volume (V0) occupied by the sample without tapping was measured after about 2 g of the powdered sample was added to a 10 ml measuring cylinder. The filled volume (V100) was read after 100 taps on the table. The bulk loose and tapped densities were computed as the weight-to-volume ratio (V0 and V100, respectively) (Ofoefule & Emeje, 2012).

$$Bulk\ density\left(\frac{g}{ml}\right) = \frac{weight\ (g)}{volume\ (ml)}$$

(2)

*Determination of Dispersibility*

1 g of native and modified starch samples were placed in a 10 ml measuring cylinder, and distilled water was added to fill to the 10 ml mark. It was thoroughly mixed and placed aside for 3 h to settle. The volume of settled particles was measured and subtracted from 10. The difference was reported as % Dispersibility according to Eqn. (3) (Ofoefule & Emeje, 2012)**.**

$$\%\ Dispersibility = 10 - volume\ of\ settled\ particle$$

(3)

*Determination of Water and Oil Absorption Capacity*

To 1 g of starch samples, 10 ml of distilled water or oil (Power Oil, Raffles Oil LFTZ Enterprise, Lagos, Nigeria) was added. The mixture was vigorously stirred for 5 min with a glass rod, it was allowed to stand for 30 min. Following that, the volume of the supernatant was recorded (Yussuf *et al.,* 2018)**.**

*Determination of Foam Capacity*

Starch samples (2 g) were homogenized in 100 ml of distilled water for 5 min using a magnetic stirrer. The homogenate was put into a 250 ml measuring cylinder after 30 s, and the volume occupied was noted. The foam capacity is expressed as a percentage increase in volume ( Isah *et al.,* 2015).

*2.6. Pasting Properties*

A paddle was put inside the canister and jostled for a few seconds before the canister was put into the Rapid Viscosity Analyzer (New Port Scientific RVA super 4). 3.5 g of starch samples were weighed. 25 ml of distilled water was then dispensed in a canister. By pressing the instrument's motor tower, the measurement cycle was initiated. The profile can be seen as it runs on the computer display attached to the instrument. The 13 min profile was employed. The temperature regime was as follows: idle temperature at 50 °C for 1 min, heated from 50 °C to 95 °C in 3 min 45 s, then held at 95 °C for 2 min 30 s, the sample was then cooled at 50 °C for 3 min 45 s, followed by a 2 min period where the temperature was controlled at 50 °C. The pasting properties of the starch sample were then displayed on a graph.

*2.7. FTIR Determination*

A 2 mg starch sample were mixed with about 200 mg of potassium bromide (KBr) and the mixture was grounded to powder. The mixture was placed in a pellet forming-die and compressed with a pressing machine to create a pellet. Following the formation of a pellet, it was moved into a cell holder and placed inside an FTIR (Perkin-Elmer Spectrum 100 FT-IR spectrometer Walth man, MA, USA) machine, and the results of the graph were displayed on the monitor.

## 3. RESULTS AND DISCUSSION

*3.1. Moisture Content (%) and Functional Properties of Native, Oxidized and Acetylated Irish Potato Starch*

**Table 1** displays the moisture content as well as the functional properties of NIPS, OIPS and AIPS. A lower moisture content ensures a longer shelf life. The % moisture content of starch indicates whether or not its water content will evaporate under identical physical conditions as water. The moisture content of NIPS, OIPS, and AIPS samples is 16.32%, 18.30%, and 18.28%, respectively. The relative humidity of the storage environment and the drying temperature have an impact on the moisture content of starch. These values are in agreement with 10-20% found in commercial starches. NIPS, OIPS, and AIPS have higher moisture contents than the values reported for Irish (14.64%) and sweet potato starch (12.03%) (Nwokocha *et al*., 2014). The pH value of NIPS, OIPS, and AIPS are 7.98, 6.41 and 6.89 respectively. The addition of an acetyl functional group into the starch molecule, which increases the acidity of the starch molecules, may explain the decrease in oxidized and acetylated starch samples pH values (Isah *et al*., 2015). These values are lower compared to 8.20-9.56 reported for white and red cocoyam starch (Yussuf *et al*., 2022). The value obtained for NIPS fall within the range described for cocoyam starch (7.19) (Jacob & Ashogbon, 2019) and 7.23 - 9.84 reported red cocoyam and cassava starch (Sanyaolu *et al.*, 2021). In starch-related industrial applications, pH is a crucial property that is typically utilized to access how alkaline or acidic liquid media are (Yussuf *et al.*, 2018; Jacob & Asogbon, 2019**).** The bulk density (Loose and tapped) of NIPS, OIPS, and AIPS falls within the range of 0.55-0.77 g/ml. The decrease in bulk density could be attributable to enhanced crystallinity as a result of chemical modification (Isah *et al*., 2015). These values obtained for NIPS and AIPS falls between the reported values of 0.71 - 0.75 g/ml for red and white cocoyam

cormel (Oladebeye *et al*., 2010) and 0.71 g/ml for cocoyam (Oladeji, 2013), less than 0.88 g/ml observed for cocoyam starch (Jacob & Asogbon, 2019) and higher than 0.39-0.50 g/ml observed for native and oxidized acha starch (Isah *et al*., 2015). Bulk density is a measurement of the weight of solid samples that can be used to determine the type of packing material required, as well as how to handle and apply materials in processing of food (Ibikunle et al., 2019). NIPS has a higher bulk density than OIPS and AIPS implying that OIPS and AIPS may find application in the food sector (Ibikunle *et al*., 2019) while NIPS may find application as an alternative binder in drug formulation and as disintegrants in pharmaceutical industry (Jacob & Asogbon, 2019). The starch dispersibility of NIPS, OIPS, and AIPS are 86.40, 87.67 and 82.13% respectively. The dispersibility of OIPS (87.67%) is higher than that of NIPS (86.40%) and AIPS (82.13%). These values fall within 85-89% reported for *Chrysophyllum albidum* kernel starch, native and phosphorylated white and red cocoyam starches (Yussuf *et al.*, 2018; Yussuf *et al*., 2022), 83.00 and 87.00% described for cassava starch blends and Bambara groundnut starch (Adeleke, 2014) and significantly higher than 40.67% described for breadfruit starch (Akanbi *et al*., 2009). The greater the dispersibility of flour, the better it reconstitutes in water. Dispersibility measures how well a flour or flour blends reconstitute in water (Yussuf *et al*., 2022; Jacob & Asogbon, 2019)**.** NIPS, OIPS and AIPS have high dispersibilities and as such they can be utilized for removing adsorptive ions in contaminated water systems, as well as in the food sector (Ihegwuagu *et al*., 2009). A substance's ability to bind with water in a limited amount of water is known as its water absorption capacity. On the other hand, dry starch ability to bind fat physically via capillary attraction is known as its oil absorption capacity. This ability is crucial because fat is used to retain flavor and improve the mouthfeel of food (Yussuf *et al.*, 2018). **Table 1** shows that the water absorption capacity (WAC) of NIPS decreased upon chemical modification while oil absorption capacity (OAC) improved upon chemical modification. NIPS had a higher WAC and a lower OAC than OIPS and AIPS. The improvement observed in the OAC after chemical modification may be due to the introduction of the functional groups onto starch molecules. An increase in OAC enhances food preparations, mouthfeel, palatability, and flavor retention ( Tharanathan, 2005; Oderinde *et al*., 2023;). OIPS and AIPS had a better foam capacity than NIPS, as depicted in **Table 1**. The values obtained for NIPS, OIPS and AIPS were 5.00, 6.50 and 6.70% respectively This result shows that NIPS have a lower fat content than OIPS and AIPS since the foam capacity is directly proportional to the fat content. The improvement in the foam capacity of OIPS and AIPS after chemical modification shows that they may be used as emulsifier in the food sector (Ihegwuagu *et al*., 2009).

**Table 1:** Moisture and functional properties of native, oxidized and acetylated Irish potato starch

| PARAMETERS | NIPS | OIPS | AIPS |
|---|---|---|---|
| Moisture content (%) | 16.32±0.00 | 18.30±0.00 | 18.28±0.00 |
| pH | 7.98±0.02 | 6.41±0.05 | 6.89±0.02 |
| Loose density (g/ml) | 0.60±0.02 | 0.55±0.15 | 0.57±0.01 |
| Tapped density (g/ml) | 0.77±0.09 | 0.68±0.01 | 0.73±0.02 |
| Dispersibility (%) | 86.40±0.33 | 87.67±0.47 | 82.13 ±0.19 |
| Water absorption capacity (%) | 266.70±0.47 | 216.70±0.24 | 103.30±0.02 |
| Oil absorption capacity (%) | 105.0±0.02 | 133.0 ± 0.10 | 250±0.01 |
| Foam capacity (%) | 5.00±0.02 | 6.50±0.05 | 6.70 ± 0.09 |

**NB:** The values are expressed on dry weight basis ± standard deviation from mean of triplicates

*3.2. Pasting properties*

**Table 2** displays the pasting properties of NIPS, OIPS and AIPS. Pasting properties explains how starch-based products and starch behave when heated in the presence of water (Olatunde *et al*., 2017). AIPS had the highest peak viscosity (1423.75 RVU), followed by OIPS (1329.33 RVU) and NIPS had the least peak viscosity (1312.33 RVU). Oxidation and acetylation increased the peak viscosity of NIPS. The increase observed in the peak viscosity of the OIPS may be because of the carboxylic groups while the higher peak viscosity following acetylation may be associated with a higher incorporation of acetyl groups, that may establish cross-links between them (Halal *et al*., 2015). The final viscosity and the setback decreased following oxidation and acetylation. NIPS (478.92 RVU) had a higher viscosity than AIPS (429.08RVU) which in turn has a higher final viscosity than OIPS (389.75 RVU). The insertion of acetyl groups is thought to have partially disrupted the granules' ability to swell, rendering the starch hydrophobic and reducing its capacity for water absorption and retention. This disruption is responsible for the decrease in the final viscosity of acetylated starch (Halal *et al*., 2015) while a reduction observed in the final viscosity of oxidized starch may be attributed to the higher oxidant concentrations which causes partial breakage of the glycoside bonds, thereby resulting in a drop in viscosity (Halal *et al*., 2015). Higher final and peak viscosities at high temperatures suggest the possibility of using them as thickeners in items that need to be sterilized, such infant food and sauces. (Olatunde *et al*., 2017). Guerra-Dellavalle *et al*., (2009) reported a similar trend in the peak viscosities of acetylated and oxidized banana starches. The higher setback values of NIPS (318.42 RVU) imply a reduced propensity for retrogradation than AIPS 231.97 (RVU) and OIPS (188.58 RVU). Oxidation decreased the breakdown viscosity while acetylation increased the breakdown viscosity of NIPS. This shows that oxidation improved the mechanical, shearing and thermal stabilities of Irish potato starch while acetylation did not (Oderinde *et al*., 2023). NIPS, OIPS and AIPS had a trough value of 160.5, 201.17 and 197.17 RVU respectively. Trough, or

holding strength can be defined as the granules' ability to withstand a period of constant mechanical shear stress and high temperature. Usually a breakdown in viscosity accompanies it (Olatunde *et al*., 2017). Peak time is the amount of time that the starch granules need to heat up to attain the maximum paste viscosity. The peak time of NIPS, OIPS and AIPS were 5.53, 3.33 and 3.33 min respectively. NIPS, OIPS and AIPS had a pasting temperature of 70.20, 70.25 and 69.35 $^{o}$C. The lower the pasting temperature, the lower the thermal energy required for breaking down the starch samples and for paste formation (Olatunde *et al*., 2017).

**Table 2:** Pasting characteristics of native and modified starch samples

| STARCH SAMPLES | PEAK VISCOSITY | TROUGH | BREAKDOWN | FINAL VISCOSITY | SETBACK | PEAK TIME | PASTING TEMP |
|---|---|---|---|---|---|---|---|
| **NIPS** | 1312.33 | 160.5 | 1151.83 | 478.92 | 318.42 | 3.53 | 70.20 |
| **OIPS** | 1329.33 | 201.17 | 1128.17 | 389.75 | 188.58 | 3.33 | 70.25 |
| **AIPS** | 1423.75 | 197.17 | 1226.58 | 429.08 | 231.97 | 3.33 | 69.35 |

*3.3. Fourier Transform Infrared Spectroscopy (FTIR)*

**Figure 1** depicts the FTIR spectra of NIPS, OIPS, and AIPS samples. Prominent characteristic broad peaks were observed at 3100 - 3600 $cm^{-1}$, strong absorption peaks between 2920 – 2950 $cm^{-1}$, 1640 – 1660 $cm^{-1}$, and 1154 - 1162 $cm^{-1}$ for NIPS, OIPS and AIPS. The IR Spectrum of NIPS showed distinct broad peaks at 3100 - 3600 $cm^{-1}$ ascribed to O–H stretch of the alcohol group, 2920 $cm^{-1}$ ascribed to C–H asymmetric Stretch, strong absorption peaks around 1636 $cm^{-1}$ were ascribed to C=O stretching mode and 1154 and 1016 $cm^{-1}$ ascribed to asymmetric C-O-C and CO vibration respectively (Oderinde *et al*., 2020; Yussuf *et al*., 2022; Oderinde *et al*., 2023). Following oxidation and acetylation, although similar overall absorption peaks were observed for both the native and the modified starches, a sharp decline or increase were observed in overall intensity. For OIPS and AIPS, the C–H asymmetric stretch absorption peak reduced to 2906 $cm^{-1}$ and 2912 $cm^{-1}$ respectively, the strong C=O stretching mode characteristic absorption peaks increased to 1672 $cm^{-1}$ and 1648 $cm^{-1}$, and the asymmetric C-O-C absorption peaks increased to 1172 $cm^{-1}$ and 1164 $cm^{-1}$ respectively. Similar observations have been observed for oxidized barley starches modified with 1%, 1.5% and 2% active chlorine (Halal *et al*., 2015) and for acetylated white and red cocoyam starches (Ibikunle *et al*., 2022). This is a confirmation that the starches were successfully modified.

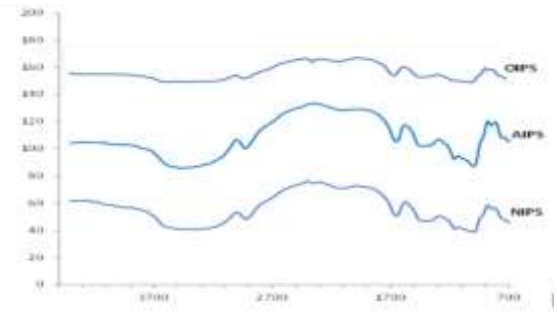


**Figure 1.0:** Infrared spectra of native, oxidized and acetylated Irish potato starch

## 4. CONCLUSION

Native Irish potato starch was chemically modified through oxidation and acetylation. The moisture content, oil absorption capacity (OAC) and foam capacity improved upon oxidation and acetylation while the bulk density, pH, and water absorption capacity (WAC) of native Irish potato starch reduced upon chemical modification. Due to enhanced OAC of NIPS by oxidation and acetylation, OIPS and AIPS can be employed as flavor retention agent. OIPS will be a preferable option when compared to NIPS and AIPS in applications such as for weaning of foods where low viscosities are desired. NIPS, OIPS, and AIPS have high peak and final viscosities, indicating their potential use as thickeners in goods that require sterilization, such as sauces and infant meals. Native, oxidized and acetylated Irish potato starch can be used to lessen staling in baked goods because they have greater setback viscosity values, which indicates a decreased susceptibility for retrogradation. In other words, native and modified Irish potato starch can be an alternative to all other conventional sources of starch for use in food, pharmaceutical and packaging industries.

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