

## Functional and textural properties of native and modified selected tropical root and tuber starches

Ifediba D I, Owuamanam C I, Omeire G C, Nwakudu A A

Federal University of Technology, Owerri, Imo State, Nigeria

Corresponding Author: Ifediba D I

### Abstract

Native starch was extracted from selected tropical root and tuber crops, namely cassava (*Manihot esculantum* TME 419), white Guinea yam (*Dioscorea rotundata* Poir.) and sweet potato (*Ipomoeo batatas* Lam.). The extracted starches were respectively chemically modified by acetylation, acid hydrolysis and oxidation using similar protocol. The starch samples were analyzed for functional and textural properties which are important indicators of starch commercial viability. There was significant ( $p < 0.05$ ) difference in the functional parameters of the native and modified starches. Acetylation generally increased the bulk density and water absorption capacities of the starch while acid hydrolysis decreased same. Modification had limited effect on the oil absorption capacity of native starches, while acid hydrolysis increased the water binding capacity and solubility more than acetylation and oxidation, while acetylation increased the swelling power more substantially. The pH of the starch samples were within the medium acidity range. There was significant ( $p < 0.05$ ) difference in the textural property of starch samples with acid hydrolysis reducing the values to indetectable minimal. The hardness, springiness and cohesiveness of samples were influenced by starch botanical source as evidenced in the dissimilar outcome of each treatment method. Modification generally reduced gumminess of the native starches, while oxidation improved their chewiness, resilience and adhesiveness more than oxidation and acid hydrolysis. Overall, modification, especially acetylation of starch, impacted positively on the functional and textural properties of the root and tuber starches under consideration.

**Keywords:** Modification, starch, textural, native, functional

### Introduction

Starch is an abundant organic compound derived from plants. It is a naturally occurring, biodegradable, inexpensive and abundant carbohydrate reserve, widely distributed in the form of tiny granules in flowers, fruits, grains, different types of stems and roots of all forms of green leafed plants (Alcazar-Alay and Meireles, 2015). It is an insoluble carbohydrate that is composed of  $\alpha$ -glucose polymers (Lozanno-Navarro *et al.*, 2018), consisting essentially of amylose and amylopectin polymers. These two major polysaccharides in starch support characteristics of biodegradable and thermoplastic polymers (Martinez-Garcia *et al.*, 2017) [28]. The glucose polymers that form starch are linked by  $\alpha$ -1, 4- and  $\alpha$ -1, 6- bonds (Li *et al.*, 2018) [30], and the  $\alpha$ -1, 4-glycosidic bonds are much easier to hydrolyze than  $\alpha$ -1, 6-glycosidic bonds (Yu *et al.*, 2018) [49]. The proportion of amylose within starch granules typically range from 20 – 30%, whereas the amylopectin composes of approximately 70 – 80% of starch of most species (Obadi and Xu, 2021; Okyere *et al.*, 2022) [37, 39]. The predominantly linear amylose has more ordered and crystalline structures that tends to form stronger gels with higher tendency to retrograde, and this can influence the texture and stability of food products (Chang *et al.*, 2021) [10]. Most commercial starches are derived from cereal grains, particularly from corn, wheat and rice, and from roots and tubers, particularly potatoes, sweet potatoes and cassava; with starch obtained from corn, wheat, potatoes and cassava in their native and modified forms accounting for 90% of the world production (Gul *et al.*, 2021) [16]. Moreover, it is estimated that approximately 75% of global starch supply to industries comes from corn (USIPA, 2014). The excellent properties of root and tuber starches, as well as the environmental benignity, easy fabrication, relative

abundance, non-toxicity, and biodegradability is receiving attention, hence spurring a large number of studies on isolation, modification, physicochemical properties and application of root and tuber crops in recent times (Al-Maqtan *et al.*, 2024; Wang *et al.*, 2024) [54]. Moreover, root and tuber starches have been categorized as gluten-free starch which is desirable for people with gluten allergy (Horstmann *et al.*, 2016) [14], and is of important consideration when considering carbohydrate source that may help to reduce prevalence of cardiovascular disease (Ariwoola *et al.*, 2017) [7]. The role of starches is based on their functional properties, mainly rheological and gelatinization properties (Ai and Jane, 2015) [4]. Although native starches have been widely used in some industries, their sub-optimal property limits their industrial food applications. Such limitations include insolubility in cold water, loss of viscosity and thickening power after cooking, low shear resistance, thermal resistance, high tendency towards retro-gradation and syneresis (Kavlani *et al.*, 2012; Korma *et al.*, 2016) [22, 23]. The common purpose of starch modification is to stabilize starch granules during processing and make it suitable for many food and industrial applications (Yan and Zhengbiao, 2010) [58]. The amylose and amylopectin content in starch is crucial when starch is modified using physical, chemical, enzymatic, biotechnological methods, or their combinations, which aim to produce starch with diverse potentials and various novel derivatives, with improvised physicochemical and structural properties. Of the four categorized methods of starch modification chemical modification of starches have recently drawn greatest attention, particularly with respect to the application of starch in the food industry (Ackar *et al.*, 2015) [13]. Chemical modification incorporates functional groups (carboxylic, ester, and amino groups) into starch

molecules, significantly conferring considerable improvements in starch molecules physicochemical properties, while retaining the molecules form and size (Obadi and Xu, 2021) [37]. Chemical modification of starch includes esterification, etherification, oxidation, acid hydrolysis and crosslinking; which are utilized to modify the molecular configuration and functional characteristics of starch, such as swelling, gelatinization and retrogradation. This can be carried out in single, dual and tertiary (multiple) folds, with the main objective to meet the specific requirement of industries ((Scott and Awika, 2023; Li, 2022; Wang *et al.* 2022) [44, 55]. Chemical modification by acetylation, acid hydrolysis and oxidation are common methods employed for starch modification for decades with profound success. This study is designed to examine the functional and textural properties of selected tropical root and tuber starches, and the effect of acetylation, acid hydrolysis and oxidation on these very important parameters that defines the commercial acceptance of starch for industrial food purposes.

## Materials and methods

### 1. Root and Tuber Materials Procurement

The cassava (*Manihot esculantum*, TME 419) roots were purchased from National Root Crops Research Institute (NRCRI) Sub-Station Farm, Igbariam, while Sweet potato (*Ipomoea batatas* Lam.) roots and White Guinea yam (*Dioscorea rotundata* Poir.) tubers were purchased from growers from Otuocha Agricultural base, both in Anambra East Local Government Area, Anambra State, Nigeria. The roots and tuber crops which were harvested at full maturity were authenticated at the Faculty of Agriculture, Chukwuemeka Odumegwu Ojukwu University, Igbariam, Anambra State, Nigeria.

## 2. Sample Production

### 2.1 Extraction of native cassava starch

The method described by Ariwaodo *et al.* (2021) [6] was used with slight modification. The fresh cassava roots were processed within 24 h of harvest from the farm to avoid biochemical degradation. The roots were peeled, washed with clean water, cut to slices and mashed in a mobile grater. The resultant cassava mash was dispersed in clean water twice its volume to obtain coarse slurry. The slurry was flushed through a 200 mesh screens with excess volume of clean water into a transparent plastic bucket to separate fibers and other extraneous matters. The filtrate was left to settle for 1 h. The supernatant was decanted and the sediment was consecutively re-suspended in distilled water twice its volume and on each occasion left to settle for 30 min before passing through 100 mesh (BSS) sieve until the supernatant became transparent. The colored top layer was manually scrapped off and the white layer was re-suspended in distilled water and left to settle as before. The process was repeated two more times to obtain a clear supernatant and stain free top layer dense paste. The firm, dense and snow-white paste was disintegrated in a shallow tray, allowed to equilibrate for 20 min under ambient conditions, and dried in air oven at 40°C for 24 h. The dried flakes was pulverized in a milling machine (CORONA Mill Model Landerx YCIA-SA, Japan), sift through 150µm screen, packed in air tight plastic container and stored under dry conditions at room temperature until use.

### 2.2 Extraction of native sweet potato starch

Potato starch was extracted using modified method of Dhiya *et al.* (2022) [12]. Fresh potato tubers were peeled, washed and reduced to shreds that were submerged in clean water to suppress oxidative browning. The potato shreds were wet-milled using a motorized mill with clean water twice its volume. The slurry was passed through a 200 mesh screens with excess volume of clean water into a transparent plastic bucket to separate extraneous matters. The filtrate was left to settle for 1 h. The supernatant was decanted and the sediment was consecutively re-suspended in distilled water twice its volume and on each occasion left to settle for 30 min before passing through 100 mesh (BSS) sieve, until the supernatant became transparent. The colored top layer was manually scrapped off and the paste was re-suspended in distilled water and left to settle as before. The process was repeated two more times to obtain a clear supernatant and stain free top layer. The firm, dense and snow-white paste was disintegrated in a shallow tray, allowed to equilibrate for 20 min under ambient conditions, and dried in air oven at 40°C for 24 h. The dried flakes was pulverized in a milling machine (CORONA Mill Model Landerx YCIA-SA, Japan), sift through 150µm screens and packed in air tight plastic container and stored under dry conditions at room temperature until use.

### 2.3 Extraction of native yam starch

Yam starch was extracted according to the method of Ezeocha and Okafor (2016) [15] with slight modification. Fresh yam tubers were peeled, washed, sliced to thin chips submerged in clean water to suppress oxidative browning. The chips were wet-milled with clean water twice its volume using a motorized mill. The slurry was flushed through a 200 mesh screens with excess volume of clean water into a transparent plastic bucket to separate extraneous matters. The filtrate was left to settle for 1 h. The supernatant was decanted and the sediment was consecutively re-suspended in distilled water twice its volume and on each occasion left to settle for 30 min before passing through 100 mesh (BSS) sieve until the supernatant became transparent. The colored top layer was manually scrapped and the pasts was re-suspended in distilled water and left to settle as before. The process was repeated two more times to obtain a clear supernatant and stain free top layer. The firm, dense and snow-white paste was disintegrated in a shallow tray, allowed to equilibrate for 20 min under ambient conditions, and dried in air oven at 40°C for 24 h. The dried flakes was pulverized in a milling machine (CORONA Mill Model Landerx YCIA-SA, Japan) to pass through 150µm screens, packed in air tight container and was stored under dry conditions at room temperature until use.

### 2.4 Modification by Acetylation

The method described by Shaari *et al.* (2021) [45] was slightly modified. 500 g (dry basis) of each of the native starches obtained from cassava, sweet potato and yam was respectively dispersed in 250 ml of distilled water and continuously stirred. The pH of the slurry was adjusted to 8.0 using 0.5M NaOH. Acetic anhydride (8g) was added drop wise to the stirred slurry, while maintaining the pH within the range of 8.0 - 8.5 using 0.5M NaOH solution. The reaction continued for 60 min before adjusting to pH4.5 with 0.2M HCL. After sedimentation, the starch slurry was

washed thrice with threefold of distilled water and once with 95% ethanol, The dense starch paste was disintegrated and spread in a shallow tray to equilibrate for 20 min under ambient conditions before drying in air oven at 40°C for 24 h. The dried flakes was pulverized in a milling machine (CORONA Mill Model Landex YCIA-SA, Japan), passed through 150µm sieve, packed in air tight plastic container and stored under dry conditions at room temperature until use.

## 2.5 Modification by acid hydrolysis

The method described by Ulbrich and Floter (2019) [51] was slightly modified. Starch hydrolysis was carried out by dispersing 500 g (dry basis) of each of the native starches obtained from cassava, sweet potato and yam in 815 mL 6% HCL solution with continuous stirring for 30 min. The reaction was allowed to stand for 10 h in a water bath at 50°C without further stirring. The slurry was neutralized with 0.5 M NaOH, washed three times with threefold volume of deionized water. The dense starch paste was disintegrated and spread in a shallow tray to equilibrate for 20 min under ambient conditions before drying in air oven at 40°C for 24 h. The dried flakes was pulverized in a milling machine (CORONA Mill Model Landex YCIA-SA, Japan), pass through 150µm sieve, packed in air tight plastic container and stored under dry conditions at room temperature until use.

## 2.6 Modification by oxidation

The method described by Wojcickowski *et al.* (2017) [28] was slightly modified. Each of the native starches obtained from cassava, sweet potato and yam was suspended in deionized water at 35% (w/w). The pH of each of the starch slurries was adjusted to 9.5 using 0.1M NaOH. Standardized sodium hypochlorite (NaOCl) solution (2g active chlorine/100g starch) was slowly added, while keeping the pH of the slurry at 9.5. After complete addition of NaOCl the slurry was maintained at the same pH for 60 min. The pH was adjusted to 7.0 with 0.1M HCL. The oxidized starch was recovered by filtration and washed with threefold of distilled water until complete removal of the ClO<sup>-</sup> ions. The dense starch slurry was disintegrated and spread in a shallow tray to equilibrate for 30 min under ambient conditions before drying in air oven at 40°C for 24 h. The dried flakes was pulverized in a milling machine (CORONA Mill Model Landex YCIA-SA, Japan), passed through 150µm sieve, packed in air tight container and stored under dry conditions at room temperature until use.

## 3. Analysis of Starch Samples

### 3.1 Functional Properties

#### 3.1.1 Bulk density (BD)

Bulk density was determined according to the method described by Adewumi *et al.* (2020) [3] with slight modification. Starch sample weighing 50 g was gently filled into a 100 mL measuring glass cylinder. The bottom of the cylinder was tapped on the laboratory bench repeatedly until there was no further diminution in sample level. The bulk density was calculated from sample weight (g) divided by final volume (mL) attained after tapping as:

$$\text{Bulk density} = \frac{W_2 - W_3}{V} \quad \text{Eqn. (1)}$$

Where W<sub>2</sub> is weight of cylinder and sample, W<sub>3</sub> is weight of empty cylinder, V is final volume attained after tapping.

#### 3.1.2 Water absorption capacity (WAC)

The method described by Odi and Sinija (2021) [38] was used. One gram (1g) of starch sample was weighed and filled into a centrifuge tube. 10 mL of water was added to the tube containing the starch sample. The suspension was homogenized for 30 min. The homogenized sample was centrifuged at 3000 rpm for 15min and the supernatant was discarded to obtain sediment. The sediment was weighed. The water absorption capacity was calculated as:

$$\text{Water absorption capacity\%} = \frac{\text{Weight of sediment}}{\text{Weight of sample}} \times 100 \quad \text{Eqn. (2)}$$

#### 3.1.3 Oil absorption capacity (OAC)

The oil absorption capacity was determined according to the method described by de Castro *et al.* (2019) with some adaptations. One gram (1g) of starch sample was weighed into a centrifuge tube and 10 mL of refined soya oil was added to the centrifuge containing the starch sample. The suspension was homogenized for 30 min and was centrifuged at 3000 rpm for 25 min. The supernatant was decanted and the weight of the resultant sediment was recorded. Oil absorption capacity was calculated as:

$$\text{Oil absorption capacity (\%)} = \frac{\text{Weight of sediment}}{\text{Weight of sample}} \times 100 \quad \text{Eqn. (3)}$$

#### 3.1.4 Water binding capacity

The method described by Omuluche *et al.* (2023) [41] was slightly modified. Five grams (5g) of starch sample was dissolved in 70 mL of distilled water with constant stirring for 1h before centrifuging at 3000rpm for 15min. The supernatant was decanted and the wet residue was allowed to drain for 20min. The residue was weighed and the weight was recorded. Water binding capacity of the samples was calculated as:

$$\text{Water binding capacity \%} = \frac{\text{Weight of sediment}}{\text{Weight of sample}} \times 100 \quad \text{Eqn (4)}$$

#### 3.1.5 Solubility (SOL)

The method of Estrada-Leon *et al.* (2016) [14] was used with slight modification. One gram (1g) of starch sample was added to 100 mL of distilled water. The suspension was heated in a water bath to 85°C following stirring at 10 min intervals for 1 h. The heated sample was cooled to 30°C. The cooled sample was transferred to a centrifuge tube and centrifuged at 2000rpm for 15 min. The supernatant was decanted into a petri dish of known weight (W<sub>1</sub>) and was dried in a hot air oven until constant weight (W<sub>2</sub>). The weight of dried solids was obtained as W<sub>2</sub> - W<sub>1</sub>. Solubility was calculated as:

$$\text{Solubility \%} = \frac{\text{Weight of dried solids}}{\text{Weight of starch sample}} \times 100 \quad \text{Eqn. (5)}$$

#### 3.1.6 Swelling power (SP)

The method described by Kaur *et al.* (2018) [9] was slightly modified. One gram (1g) of starch sample was added to 100 mL of distilled water. The dispersion was heated in a water bath at 60°C, 70°C, 80°C and 90°C with constant stirring for

1 h. The heated sample was cooled to 30°C. The cooled sample was transferred to a centrifuge and centrifuged at 2000 rpm for 10 min. The supernatant was decanted and the sediment was weighed. The supernatant was dried in a hot air oven and the dried solids were weighed. The swelling power was calculated as:

$$\text{Swelling power} = \frac{\text{Weight of sediment}}{\text{Weight of sample} - \text{Weight of dried solids}} \times 100 \quad \text{Eqn. (6)}$$

### 3.1.7 pH

The method described by Omuluche *et al.* (2023) [41] was used with slight modification. Starch sample weighing 2 g was dissolved in 10 mL of distilled water with careful stirring with glass rod for 5 min. The pH was determined using a digital pH meter.

### 3.2 Textural Properties of Starch Gels

The method described by Bajaj *et al.* (2018) [9] was slightly modified. 2% starch suspension (w/w) was prepared. The suspension was heated in a water bath at 95°C for 30 min. The gels were stored in tubes under refrigeration conditions at 4°C for 24 h. The texture profile analyses (TPA) of stored gels was conducted using texture analyzer (TAXT-2i, Stable Micro Systems, UK) equipped with 1 kg load cell, compressed at a speed of 0.5 mm/s to a distance of 10 mm

with a cylindrical plunger of diameter 5 mm. The compression was repeated twice to generate force time curve from which hardness (height of first peak) and springiness (ratio between recovered height after the first compression and the original gel height) were determined. Adhesiveness was taken as the negative area of the curve during retraction of the probe. Cohesiveness was calculated as the ratio between the area under the second peak and the area under the first peak. Gumminess was determined by multiplying hardness and cohesiveness, resilience was a measure of how the starch recoiled to regain the original shape, while chewiness was determined by multiplying gumminess and springiness.

### 4. Experimental design and Statistical analysis

The experimental design for the study is a 3x3 factorial experiment (with triple replication) comprising of three tropical tuber/root (cassava, sweet potato and yam), and treatments (acetylation, acid-thinning and oxidation). The data was analyzed using IBM SPSS Version 22 using One Way Analysis of Variance (ANOVA) for Multiple Comparison Test. The means and standard deviations of triplicate data values were separated for significant at 95% confidence level difference using Turkey's Test.

**Table 1:** Functional property of starch samples

| SAMPLE | Parameter                 |                          |                          |                            |                           | %SP                       | pH                        |
|--------|---------------------------|--------------------------|--------------------------|----------------------------|---------------------------|---------------------------|---------------------------|
|        | BD(g/mL)                  | WAC(g/g)                 | OAC(g/g)                 | WBC(g/g)                   | %SOL                      |                           |                           |
| NCS    | 0.830±0.014 <sup>b</sup>  | 0.750±0.014 <sup>g</sup> | 1.215±0.007 <sup>a</sup> | 77.798±0.026 <sup>bc</sup> | 19.140±0.028 <sup>j</sup> | 22.875±0.007 <sup>g</sup> | 5.545±0.007 <sup>d</sup>  |
| ACS    | 0.905±0.007 <sup>a</sup>  | 0.865±0.007 <sup>f</sup> | 1.055±0.021 <sup>b</sup> | 78.094±0.040 <sup>b</sup>  | 11.465±0.035 <sup>l</sup> | 29.435±0.035 <sup>f</sup> | 5.250±0.007 <sup>de</sup> |
| HCS    | 0.805±0.007 <sup>bc</sup> | 0.500±0.000 <sup>j</sup> | 0.965±0.021 <sup>c</sup> | 75.661±0.052 <sup>d</sup>  | 20.830±0.014 <sup>i</sup> | 20.045±0.035 <sup>h</sup> | 5.510±0.014 <sup>d</sup>  |
| OCS    | 0.700±0.000 <sup>c</sup>  | 1.180±0.014 <sup>d</sup> | 1.225±0.007 <sup>a</sup> | 76.268±0.021 <sup>c</sup>  | 24.755±0.035 <sup>d</sup> | 15.955±0.021 <sup>l</sup> | 5.705±0.007 <sup>b</sup>  |
| NPS    | 0.775±0.007 <sup>bc</sup> | 0.625±0.007 <sup>h</sup> | 0.760±0.014 <sup>d</sup> | 58.206±0.026 <sup>k</sup>  | 23.850±0.042 <sup>g</sup> | 50.385±0.035 <sup>c</sup> | 5.810±0.014 <sup>a</sup>  |
| APS    | 0.855±0.007 <sup>b</sup>  | 1.250±0.014 <sup>b</sup> | 0.930±0.014 <sup>c</sup> | 70.950±0.018 <sup>h</sup>  | 22.935±0.021 <sup>f</sup> | 60.340±0.028 <sup>c</sup> | 5.250±0.007 <sup>d</sup>  |
| HPS    | 0.725±0.007 <sup>c</sup>  | 0.585±0.007 <sup>i</sup> | 0.700±0.000 <sup>e</sup> | 77.468±0.023 <sup>bc</sup> | 29.040±0.028 <sup>b</sup> | 41.735±0.021 <sup>d</sup> | 5.555±0.007 <sup>cd</sup> |
| OPS    | 0.605±0.007 <sup>e</sup>  | 1.475±0.007 <sup>a</sup> | 0.980±0.014 <sup>c</sup> | 72.858±0.039 <sup>g</sup>  | 31.145±0.035 <sup>a</sup> | 19.160±0.014 <sup>j</sup> | 5.581±0.027 <sup>cd</sup> |
| NYS    | 0.695±0.007 <sup>c</sup>  | 0.865±0.007 <sup>f</sup> | 0.555±0.007 <sup>f</sup> | 59.458±0.080 <sup>j</sup>  | 21.725±0.021 <sup>h</sup> | 19.795±0.049 <sup>i</sup> | 5.195±0.007 <sup>de</sup> |
| AYS    | 0.780±0.014 <sup>bc</sup> | 0.925±0.007 <sup>e</sup> | 0.545±0.007 <sup>f</sup> | 67.986±0.037 <sup>i</sup>  | 16.745±0.035 <sup>k</sup> | 55.040±0.028 <sup>b</sup> | 5.465±0.007 <sup>de</sup> |
| HYS    | 0.660±0.000 <sup>d</sup>  | 0.525±0.007 <sup>j</sup> | 0.515±0.007 <sup>f</sup> | 79.806±0.019 <sup>a</sup>  | 24.135±0.021 <sup>c</sup> | 33.470±0.014 <sup>c</sup> | 4.905±0.007 <sup>c</sup>  |
| OYS    | 0.555±0.007 <sup>f</sup>  | 1.380±0.014 <sup>c</sup> | 0.685±0.007 <sup>e</sup> | 74.456±0.001 <sup>f</sup>  | 27.360±0.014 <sup>c</sup> | 16.815±0.007 <sup>k</sup> | 5.795±0.007 <sup>a</sup>  |

Means and standard deviation of triplicate values with different superscripts within a column are significantly (p<0.05) different. NCS (native cassava starch), ACS (acetylated cassava starch), HCS (hydrolyzed cassava starch), OCS (oxidized cassava starch), NPS (native sweet

potato starch), APS (acetylated sweet potato starch), HPS (hydrolyzed sweet potato starch), OPS (oxidized sweet potato starch), NYS (native yam starch), AYS (acetylated yam starch), HYS (hydrolyzed yam starch), OYS (oxidized yam starch)

**Table 3:** Textural properties of starch samples

| Sample | Parameter                  |                        |                          |                             |                            |                         |                           |
|--------|----------------------------|------------------------|--------------------------|-----------------------------|----------------------------|-------------------------|---------------------------|
|        | Hardness                   | Springiness            | Cohesiveness             | Gumminess                   | Chewiness                  | Resilience              | Adhesiveness              |
| NCS    | 398.59±47.75 <sup>e</sup>  | 1.61±1.11 <sup>a</sup> | 0.94±0.027 <sup>ab</sup> | 375.74±54.87 <sup>c</sup>   | 635.40±50.92 <sup>b</sup>  | 0.73±0.034 <sup>b</sup> | -3.98±1.00 <sup>ab</sup>  |
| ACS    | 268.09±28.96 <sup>e</sup>  | 0.97±0.03 <sup>a</sup> | 0.94±0.02 <sup>ab</sup>  | 252.69±32.64 <sup>c</sup>   | 245.47±32.55 <sup>c</sup>  | 0.700±0.02 <sup>c</sup> | -37.85±7.32 <sup>d</sup>  |
| HCS    | ND                         | ND                     | ND                       | ND                          | ND                         | ND                      | ND                        |
| OCS    | 352.99±6.49 <sup>e</sup>   | 0.99±0.01 <sup>a</sup> | 0.97±0.00 <sup>a</sup>   | 342.09±9.63 <sup>c</sup>    | 338.78±13.75 <sup>c</sup>  | 0.82±0.04 <sup>a</sup>  | -12.50±0.33 <sup>bc</sup> |
| NPS    | 566.3±22.60 <sup>d</sup>   | 0.97±0.01 <sup>a</sup> | 0.97±0.01 <sup>a</sup>   | 551.13±26.71 <sup>d</sup>   | 535.77±29.01 <sup>b</sup>  | 0.83±0.01 <sup>a</sup>  | -4.87±4.83 <sup>ab</sup>  |
| APS    | 480.99±33.54 <sup>de</sup> | 0.99±0.01 <sup>a</sup> | 0.96±0.02 <sup>a</sup>   | 463.69±39.62 <sup>d</sup>   | 459.28±35.87 <sup>b</sup>  | 0.78±0.03 <sup>ab</sup> | -9.48±1.23 <sup>b</sup>   |
| HPS    | ND                         | ND                     | ND                       | ND                          | ND                         | ND                      | ND                        |
| OPS    | 764.56±31.97 <sup>c</sup>  | 0.98±0.01 <sup>a</sup> | 0.96±0.01 <sup>a</sup>   | 735.85±41.39 <sup>c</sup>   | 724.60±48.71 <sup>a</sup>  | 0.81±0.02 <sup>ab</sup> | -14.20±4.81 <sup>c</sup>  |
| NYS    | 1389.02±56.50 <sup>a</sup> | 0.97±0.01 <sup>a</sup> | 0.92±0.02 <sup>ab</sup>  | 1282.67±75.40 <sup>a</sup>  | 1239.56±80.58 <sup>a</sup> | 0.73±0.01 <sup>b</sup>  | ND                        |
| AYS    | 1155.31±90.53 <sup>b</sup> | 0.95±0.04 <sup>a</sup> | 0.89±0.03 <sup>b</sup>   | 1034.30±124.90 <sup>b</sup> | 983.42±152.02 <sup>a</sup> | 0.67±0.04 <sup>c</sup>  | ND                        |
| HYS    | ND                         | ND                     | ND                       | ND                          | ND                         | ND                      | ND                        |
| OYS    | 1178.50±45.05 <sup>b</sup> | 0.98±0.03 <sup>a</sup> | 0.93±0.01 <sup>ab</sup>  | 1102.39±58.86 <sup>b</sup>  | 1086.03±85.35 <sup>a</sup> | 0.75±0.02 <sup>b</sup>  | -1.29±0.65 <sup>a</sup>   |

Means and standard deviation of triplicate values with different superscripts within a column are significantly ( $p < 0.05$ ) different. ND is not detected, NCS (native cassava starch), ACS (acetylated cassava starch), HCS (hydrolyzed cassava starch), OCS (oxidized cassava starch), NPS (native sweet potato starch), APS (acetylated sweet potato starch), HPS (hydrolyzed sweet potato starch), OPS (oxidized sweet potato starch), NYS (native yam starch), AYS (acetylated yam starch), HYS (hydrolyzed yam starch), OYS (oxidized yam starch)

## 1. Functional Properties

The result of functional property of starch samples is presented in Table 1.

There was significant ( $p < 0.05$ ) difference in the bulk density of starch samples. Modification treatments had similar effects, with acetylation increasing the bulk density values, while acid hydrolysis and oxidation decreased it. The bulk densities of the native starches compared with the values obtained for similar root and tuber starches (Ezeocha and Okafor, 2016; Adewumi *et al.*, 2020; Oni *et al.*, 2020) [3, 15, 42]. Variation in bulk density values can be influenced by difference in particle size and fiber content, with lower particle size leading to lower bulk density (Odi and Sinija, 2021) [38], and less fiber particles leading to higher bulk density (Huang *et al.*, 2010) [18]. The observed increase in bulk density after acetylation is consistent the reported increase in acetylated cassava and yam starches by Oni *et al.* (2020) [42]. The observed decrease in bulk densities of acid treated starches conforms to the reduction of bulk densities of acid hydrolyzed cassava and yam starches by Adewumi *et al.* (2020) [3] and the reduction in the bulk density of acid hydrolyzed achi grain starch by Isah (2018) [30]. The observed decrease in the bulk densities of the oxidized starches is consistent with Isah (2018) [30] who reported similar decrease in oxidized achi grain starch, which may be due to increase in crystallinity induced by chemical modification. Bulk density is a good index of structural changes, and the higher value in starch implies better blending and expendability properties of the starch (Navaf *et al.*, 2020) [32]. Materials with higher bulk densities are regarded as heavy, and the higher the bulk density of a material the more the quantity that can be packaged in a confined container in a various unit operations, storage and packaging, which makes it preferable (Odi and Sinija, 2021) [38]. There was significant ( $p < 0.05$ ) difference in the water absorption capacity of the starch samples. The higher water absorption capacity of yam starch than the cassava starch agrees with Oni *et al.* (2020) [42] who found similar results for cassava and yam starches. The significant increase in the water absorption capacity of acetylated starches is similar to the increase in acetylated cocoyam and yam starches by Adewumi *et al.* (2020) [3], cassava, potato starches and white yam (Mendoza *et al.*, 2016; Uwein and Itah, 2019) [29, 53]. The increase may be due to substitution of acetyl groups with possible increase in water absorbing sites in the starch granules. Omuluche *et al.* (2023) [41] noted that the increase may be due to substantial hydrogen bonding between water molecules and starch hydroxyl groups, and loose contact between amylose and amylopectin polymers in starch granules. Acetylation makes starch more lipophilic by introducing lipophilic groups into the starch, weakening the molecular bonds that hold the groups together and thus change the shape and size of granules as well as other

functional properties of starch (Acker *et al.* 2015) [2]. The decrease in the water absorption in acid hydrolyzed starch samples is consistent with the findings for cassava, yam and achi grain starches (Isah, 2018; Oni *et al.*, 2020) [30, 42]. The decrease may be due to thinning of the starch granules to smaller fiber particles which may lower the ability of fiber to absorb water (Huang *et al.*, 2010) [18]. The increase in water absorption capacity of oxidized starch is similar to the increase in achi grain starch (Isah, 2018), potato starch (Neraj and Bisht, 2018) [30, 34] and cassava starch (Satmalawati *et al.*, 2020) [33]. Water absorption capacity reflects the amount of water that starch can hold in relation to its weight (Awoyale *et al.*, 2016) [14]. Water absorption capacity is an indication of its moisture stability, which is very important in the food industry. The oil absorption capacity significantly ( $p < 0.05$ ) differed with the oxidized starches having higher values than the native and other modified starches. Acid hydrolysis decreased the oil absorption capacity in all the starches, while acetylation decreased the oil absorption capacities of cassava and yam starches but increased the value for sweet potato starch. The recorded increase in oil absorption capacity of oxidized starches is similar to the findings by Isah (2018) [49] for oxidized achi grain starch, and oxidized sweet potato starch by (Neraj *et al.*, 2020) [33]. The increase may be due to the incorporation of carbonyl and carboxyl functional groups into the starch molecules which confers hydrophilic and hydrophobic attributes on the starch granules. The reduction in the oil absorption capacity of acid modified starches differed from the reported increase in oil absorption capacity of acid treated cassava and yam starches (Oni *et al.*, 2020) [42]. The contrast may be due to difference in cultivar, maturity, starch extraction protocol, slurry concentration and degree of hydrolysis. The observed increase in the oil absorption capacities of acetylated sweet potato starches conforms with reported increase for acetylated cassava, cocoyam and yam starches (Adewumi *et al.*, 2020; Oni *et al.*, 2020) [3, 42]. There was significant ( $p < 0.05$ ) difference in water binding capacity with varied effect of modification treatment on the starch source. The vagary may be due to difference in botanical course, genotype, agronomic factors, processing and analytical conditions. Zuluaga *et al.* (2007) [63] remarked that water binding capacity is affected by the presence of minerals like phosphorus, with high levels of phosphorus inducing high water binding capacity. The higher water binding capacity of acetylated starches is in agreement with the reports that acetylation increased the water binding capacity of native starches (Uwen and Itah, 2019; Omuluche *et al.*, 2023) [41, 53]. The increase in water binding capacity may be due to variation in water binding sites within the starch granules (El-Halal *et al.*, 2015) [13], which may be attributed to substantial hydrogen bonding between water molecules and starch hydroxyl groups, and the loose contact between amylose and amylopectin in starch granules (Omuluche *et al.*, 2023) [41]. The amount of water taken up by starch is very important in the use of starch in food industries, and modification treatments that introduce more hydrophilic groups in the starch increase water binding capacity (Zhang *et al.*, 2005) [62]. There was significant ( $p < 0.05$ ) difference in the solubility of starch samples with the oxidized sample being the most soluble followed by acid treated starches, with a decrease in acetylated starches. The increased solubility of acid hydrolyzed starch conforms to the reported

increase in solubility of acid treated sweet potato starch (Moorthy *et al.*, 2016), cassava starch (Dihya *et al.*, 2022)<sup>[12, 14]</sup> and potato Nano starch (Mojo-Quisani *et al.*, 2024). The increase in the solubility of acid treated starches may be due to strong disruption of the biopolymer caused by breakdown of the amylopectin at  $\alpha$ -1,6 bond to yield lower particle size with increased solubility (Harasyin *et al.*, 2020). The observed increase in the solubility of oxidized starches agrees with previous reports that oxidation of potato and yam starch significantly increased their water solubility (Moorthy *et al.*, 2016; Neraj and Bisht, 2018; Nuswantari, 2022)<sup>[14, 34, 35]</sup>. The increase may be due to the weakening of the hydrogen bonds in the starch granules, causing the amorphous regions to stretch more, absorb more water and solubilize more easily (Surmadion *et al.*, 2019). Starch with high solubility is used in industries in the manufacture of items such as noodles, jelly and baked products (Marta *et al.*, 2022; Wang *et al.*, 2022)<sup>[27, 55]</sup>. There was significant ( $p < 0.05$ ) difference in the swelling power of the starch samples, with acetylation increasing the swelling power of the native starches while acid hydrolysis and oxidation decreased the swelling power of the native starches. Swelling power indicates the amount of water uptake at certain temperature, and the higher swelling power of native potato starch is understandable since sweet potato has higher swelling power than grain and many non-grain starch (Obadi and Xu, 2021)<sup>[37]</sup>. The increase in swelling power of acetylated starches complies with similar increase in acetylated starches of different sources as in Oni *et al.* (2020)<sup>[42]</sup> for cassava and yam starches, which may be due to weakening and disruption of intermolecular hydrogen bonds in the starch, which may increase the accessibility of starch granules to water (Olu-Owolabi *et al.*, 2014; Singh *et al.*, 2019; Dihya *et al.*, 2022; Nusawantari, 2022)<sup>[12, 40, 47]</sup>. The decrease in the swelling power of oxidized starches is consistent with the findings of Surmadiono *et al.* (2018)<sup>[49]</sup> that oxidation with ozone at pH10 decreased the swelling power of native starch. The decrease in swelling power of acid hydrolyzed starch is in agreement with reported decrease in swelling power of acid hydrolyzed achi grain starch by Isah (2018)<sup>[30]</sup>. The decrease may be attributed to the cleavage of the intermolecular bonding by acid hydrolysis, which limits the hydrogen bonding of water molecules with the hydroxyl group of the starch molecule (Estrada-Leon *et al.*, 2016)<sup>[14]</sup>. Higher swelling power is an indication of high digestibility and improved dietary attributes (Nuwamanya *et al.*, 2010). There was significant ( $p < 0.05$ ) difference in the pH of starch samples. The pH values of native starches of 5.195 to 5.810 compares with the 5.350 for cassava starch reported by Odi and Sinija (2021)<sup>[38]</sup> and 5.17 and 6.15 for cassava and sweet potato native starches by Hundekari and Swami (2023)<sup>[19]</sup>. Acid hydrolysis decreases the pH of the three native starches, which may be due to introduction of new functional groups that increases acidity. The increase in acidity after acid hydrolysis is in agreement with decrease in the pH value of sweet potato starch (Achor and Umar, 2015), and acha starch by Isah (2018)<sup>[1, 30]</sup> after acid hydrolysis. Acetylation decreased the pH of cassava and sweet potato starches but increased the pH of yam starch. This is understandable since pH is influenced by a number of factors, including botanical source and variety (Ariwaodo *et al.*, 2021)<sup>[6]</sup>. Oxidation treatment increased the pH of cassava and yam starches but decreased the value of sweet potato starch. The increase in

pH values of cassava and yam starches may be due to degradation of acidic components in the starch, or presence of basic residual oxidation byproducts as may be influenced by starch botanical source. The pH of a product is a good quality indicator because starch with pH of 4 or less will have characteristic sour aroma and taste as often caused by fermentation (Ariwaodo *et al.*, 2021)<sup>[6]</sup>. Interestingly, the pH of the starch samples fall within the pH of 3 to 9 obtained for most starches used in pharmaceutical, domestic and food industries (Adewumi *et al.*, 2020)<sup>[3]</sup>.

## 2. Textural Properties of Starch Samples

The result of the textural properties of the starch samples is presented in Table 2.

There was significant ( $p < 0.05$ ) difference in hardness of starch samples gels with yam starch being the hardest, followed by the sweet potato starches. The difference may be due to variations in starch biopolymer ratio and cellular diversity, which may react differently to modifying reagents. Hussaini *et al.* (2018)<sup>[20]</sup> found that substitution of chickpea starch in wheat starch increased gel hardness, which suggests that harder starch-water systems is directly correlated with its amylose content. This was corroborated by Talbot-Walsh *et al.* (2018) and Nasiri *et al.* (2022)<sup>[31, 50]</sup> who noted that replacement of fat with potato starch in processed cheese led to amylose leaching into the cheese matrix from starch granules, stretching the hydrogen bonds between the protein and carbohydrate molecules and increasing hardness, while low amylose starch such as waxy maize or rice starch yielded softer products in the production of imitation cheese. Sukhija *et al.* (2016)<sup>[14]</sup> stated that the starch forming harder gels tend to have higher amylose content and long amylopectin chains, and mechanical properties of starch gels depends on the amylose matrix, the volume fraction, interactions between dispersed and continuous phase of native starch granules. However, the harder yam starch gel compared to the cassava starch with higher amylose as found in this study suggests that amylose may not be sole determinant of starch hardness. It is possible that amylose-amylopectin interaction, starch source, granular characteristics, starch concentration, pasting temperatures can be other contributing factors to starch gel hardness. Acetylation generally led to a decrease in hardness in the native starches which is consistent with the report by Zhang *et al.* (2023)<sup>[61]</sup> that gel parameters of acetylated starches were lower than those of native chick pea starch. The decrease in gel hardness may be due to the introduction of acetyl functional group which severe the binding forces within the particles and weaken the hydrogen bonds. Zdybel *et al.* (2021)<sup>[60]</sup> contends that the strength of the gel depends on the binding force within the particles, which adjudged to be closely related to the content of amylose in the starch (Zhang *et al.*, 2023)<sup>[61]</sup>. Acid hydrolysis resulted in diminution of starch hardness in all the native starches as supported by Wang *et al.* (2017)<sup>[56]</sup> who found that the hardness of potato starch gels decreased with acid amino acids, but increased with basic amino acids. Sharaf *et al.* (2020) defined hardness as the force required to achieve a defined level of deformation which implies that the acid modified starches are not suitable for products requiring resilience during storage and transportation. There was marginal ( $p > 0.05$ ) difference in springiness of starch samples, indicating that starch source and treatment had minimal impact on springiness which is similar to the

findings by Wang *et al.* (2017) [56], baring degradation of molecular structure as in the acid hydrolyzed starches. There was marginal ( $p>0.05$ ) difference in the cohesiveness of starch samples with the exception of acid thinned starch which showed no detectable gel textural parameters. The strength of the internal bonds that comprise the products body is known as cohesiveness (Parra-Ocampo *et al.*, 2020) [43]. Cohesiveness has been attributed to the higher degree of polymerization of amylose in starch which renders it more cohesive (Hussain *et al.*, 2018) [20]. Moderate cohesion is ideal for easy slicing or shredding, with reduced matting. There was significant ( $p<0.05$ ) difference in gumminess of starch samples, variant effect of modification treatments. Gumminess of gel is a hardness and cohesiveness dependent parameter (Hussain *et al.*, 2018) [20], which may be reason for the high gumminess of the yam starches. Since gumminess is a product of hardness and cohesiveness, the yam starch will likely provide gels with more rigid and firm textures which is desirable in products like custard and puddings. There was significant ( $p<0.05$ ) difference in the chewiness and resilience of starch samples. The chewiness values of 375.74, 551.13 and 1239.56 for native cassava, potato and yam starches respectively were largely lower than the 960.37, 2116.57 and 3195.85 of wheat, Turkish bean and chickpea starches (Hussain *et al.*, 2018) [20], suggesting that grains produces starches with higher chewiness than roots and tubers. There was significant ( $p<0.05$ ) difference in the resilience of the starch samples with the yam starch leading as in the other textural parameters. Resilience is a measure of how a starch rebounds to regain its original shape immediately after the first compression. It is governed by the starch gel network as influenced by amylose/amylopectin ratio, retrogradation, granular integrity, and interactions with water, protein and fat. The higher the resilience the more the elastic recovery, suggesting that the sweet potato starches may exhibit higher elastic recovery than the cassava and yam starches. The starches significantly ( $p<0.05$ ) differed in adhesiveness, with acid thinned samples consistently indicating poor textural results.

## Conclusion

Tropical root and tuber crops are tremendous repository of starch a relative ease of extraction due to limited fat content. Native starches has certain drawbacks to its versatile application in food and non-food systems, and this can be addressed through appropriate modification treatment. The efficacy of acetylation, acid hydrolysis and oxidation in retuning the deficiencies of native starch is manifest in the improved functional and textural properties of the tropical root and tuber starches selected for this study. Further researches are require to explore treatment variables that yield optimal results with the view to obtain starch products tailored to meet the growing demand of starch at the local and international levels, with the expected boost to the economy.

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