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Influence of Chemical Modification on Some Properties of Starches from Tiger Nut (*Cyperus esculentus*) and Cocoyam (*Xanthosoma sagittifolium*) as a Potential Biomaterial

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ABSTRACT

Starch is known to be the principal carbohydrate constituent of many plant materials and this necessitate a detailed investigation to understand better its biochemical and functional characteristics because they are used by the food, paper, chemical, pharmaceutical, textile and other industries. In this study, starches were extracted from cocoyam and tiger nut using standard methods and were modified by oxidation, acetylation and pre-gelatinization and evaluated for chemical, functional, pasting properties and colour. The result shows that pre-gelatinization significantly ($p<0.05$) increases the solubility, viscosity and paste clarity of tigernut and cocoyam starches by 12.5 and 10.33%, 1065 and 2188.33 CP, 16.31% T and 23.33%T, respectively. Modification significantly ($p<0.05$) increases the water holding capacity of tigernut by 2.00% (acetylation), dual modification (1.77%), oxidized starch (1.55%). The water holding capacity of both the dual and modified tiger nut starches had no significant difference ($p<0.05$). Pre-gelatinization improves the swelling and solubility properties of cocoyam starch which was required in baking industry and also improved the viscosity which is useful in adhesive industries where high viscosity is required. Dual modified tigernut and cocoyam had high OAC of 0.75 and 1.07%, respectively, which was of great importance as fat acts as flavor retainer and increases the mouth feel of food. In this report, it shows that pregelatinized starch possessed the highest L* value (108.34) and the lowest chrome C* value (12.44), therefore it is desirable to meet consumer's preference. From these results concluded that the undesirable properties in native cocoyam and tigernut starches could be overcome through the use dual-modification and pregelatinization.

Key words: Starch, pre-gelatinization, oxidation, acetylation, dual-modification, chemical composition, functional properties

INTRODUCTION

Starch or amylum is a polysaccharide carbohydrate consisting of a large number of glucose unit joined together by glycosidic bonds. It comprises of amylose and amylopectin as macromolecule. It was produced by all green plants as an energy store and is an important energy source for all human. It is found in potato, wheat, rice and other foods and it varies in appearance, depending on its source[1]. The main location of starch synthesis and storage in cereals is the endosperm. Major starch sources are cereals (40-90%), roots (30-70%), tubers (65-85%), legumes (25-50%) and some immature fruits like bananas, mangos, which contain approximately 70% of starch by

dry weight[2]. Starch has several influences in food which may include moisture, constituent and shelf stability. It may also attract or inhibit moisture in foods. Starch is one of the most important products in the world; an essential component of food, providing a large proportion of daily calorie intake for both humans and livestock. Starch alone accounts for 60-70% of calorie intake of humans[3]. Besides its nutritive value, starch is a very versatile raw material with a wide range of applications in food, feed, pharmaceutical, textile, paper, cosmetic and construction industries. Starch may also be used as a thickener, filler which may increase the solid content of soups, as a binder to consolidate the mass of food and prevent the food from drying out during cooking and as a stabilizer in the food industry[4]. Starch has also found applications as adhesives in the paper and packaging industry, match-head binders in explosives and also in construction industry as concrete block binders and plywood adhesive; fabric finishing and printing in the textile industry while as pill coating and dispersing agents in pharmaceuticals.

It has also found application as sintered metal adhesive and foundry core binders in metals and manufacture of biodegradable plastics and dry cell batteries[5]. These applications vary considerably from one botanical source to another and are dependent on some starches properties such as gelatinization, pasting, retrogradation, water absorption capacity, swelling power and solubility[5]. The physiochemical properties of most starches were a function of the composition and proportion of amylose and amylopectin in the starches[6]. Despite their advantageous properties, some starch in their native form may pose problems. For example, the tendency of its viscosity to increase rapidly and be thickened during heat may cause difficulties in industrial food unit operations such as in pumps and heat exchangers. Starch in an unmodified form has limited use in food industry because the native starch granules hydrate easily, swell rapidly, rupture, loose viscosity and produce weak bodied with very stringy and cohesive paste[7]. Starch is partially modified by degrading the starch in order to take of all aforementioned problems by meeting the end-user requirement to produce several specialty products[8]. The purpose of these modifications were to enhance its properties by improving the water holding capacity, heat resistance behavior, reinforce its binding, minimizing syneresis of starch and improve thickening[9,10]. Modification does not alter the appearance of starch but only improve the desired properties of starch.

Therefore, tiger nut and African yam bean being an underutilized crop and legume respectively will be of more importance in food industries and pharmaceutical industries. Tigernut (*Cyperus esculentus*, Linn. belongs to the family *Cyperaceae* and is a juicy and sweet nut. The crop has not enjoyed maximum patronage owning to wide consumption as a junk food or at most as a snack. Although, it is still underutilized but it is general enjoyed by many people because of its palatability and it is known to be rich in starch, oil, minerals and vitamins E and C[11,12]. It was also a good source of phosphorus, potassium, iron, magnesium, calcium, zinc, copper, sodium and manganese[13]. It has been reported to be high in dietary fibre content which could be used in the treatment and prevention of many degenerative diseases such as colon cancer, coronary heart diseases, obesity, diabetes and gastrointestinal diseases[14].

Cocoyam (*Xanthosoma sagittifolium*) was an underutilized crop that belongs to the family *Araceae;* and was referred to as cocoyam in many part of the world, especially in Africa, where they were mainly grown for their edible starch storage corms and cormels which are called tubers and also as a leafy vegetable[15]. It is very rich in vitamin B_6 and magnesium which help to prevent high blood pressure and protect the heart. It is very rich in dietary fibre and good source of glucose[16]. Starch in their natural state when use in food industries tends to produce weak-bodied, cohesive, rubbery paste when heated and undesirable gel when paste is cooled. Therefore, when modified it serve as additives and also improve the physiochemical properties and qualities of the starch. Similar work had been done by Ganiyat *et al.*[17] on banana and plantain starch which was modified by acetylation, oxidation and pre-gelatinization which reduces the peak, trough, final and setback viscosity of banana and plantain starch but increases the breakdown viscosity of banana starch. Native starches have less application because of their inability to tolerate processing conditions with the following drawbacks: low paste clarity, low sensitivity to pH, low heat and shear, high retrogradation, low decomposition and syneresis, hence there is need to improve these desirable functional properties. These drawbacks may overcome by modifying the starches through several processes which may include: physical (gamma irradiation and heat-moisture treatment), chemical (acetylation, acid-thinned, oxidation, cross-linking and esterification) and enzymatic process[18]. Therefore this research study on tiger nut starch and cocoyam starch will help suggest their potential uses in food processing industries and in pharmaceutical industries since they are being seen as underutilized crop and legume respectively and in areas where more attention are not given to them, particularly in terms of their health benefit. This lack of knowledge has limited the use of starch from these crops in various industrial applications. Hence, physicochemical, functional and structural properties of the two crops need to be investigated

and evaluated so as to consider them as new sources of starch for food and pharmaceutical industries in Nigerian. Such knowledge would unravel the opportunities offered by these crops and facilitate the utilization of starches from these crops in the industry. Furthermore, a detailed knowledge of these starch characteristics would improve and enhance their properties by physical and/or chemical modification and help Nigeria to compete effectively on the markets. In the long run, utilization of these starches will save foreign currency, create employment opportunities and bring economic benefit to the local Nigerians. The objective of this study was to evaluate chemical, functional and pasting properties of tigernut and cocoyam starches modify by acetylation, oxidation and pre-gelatinization.

MATERIALS AND METHODOLOGY

Materials: Tigernut and Cocoyam were purchased at Oyingbo Market, Lagos State.

Starch isolation: Starches were extracted from tiger nut and cocoyam using the modified method of Alabi *et al.*[16] as shown in Fig. 1 and 2. The sorted (530 g) were washed and steeped in sodium metabisulphite solution (0.81339 $Na_2S_2O_5$ L^{-1}) at 27°C for 48 h. The steeped water was changed after 24 h, then the tubers were milled into slurry which was suspended in 2 L of $Na_2S_2O_5$ solution, stirred and allowed to stand for 2 min, afterwards the starch milk was stirred again, passed through a 100 µ mesh sieve cloth and the suspension was allowed to stand for 24 h. The supernatant was decanted and the starch sediment was collected and re-suspended in distilled water. The starch milk was passed through a 250 µ sieve and then centrifuge at 3000 rpm for 20 min. The wet cake was crushed manually, dried at room temperature for 24 h and then oven dried at 50°C for 3 h. Then it was thoroughly milled, sieved, packaged in polythene bags and stored at ambient temperature (27±2°C).

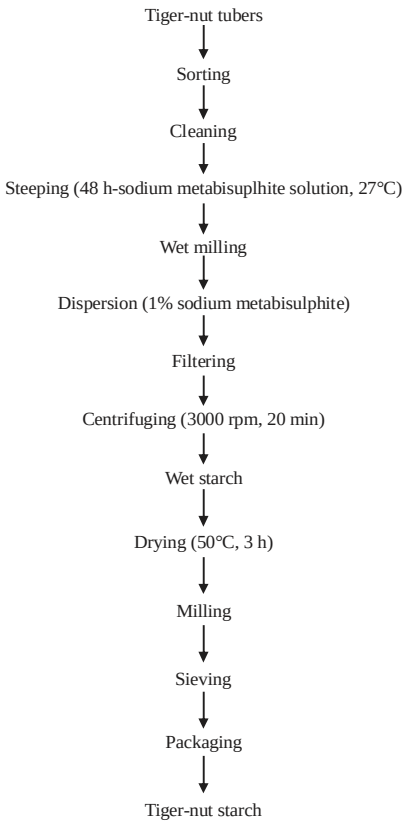

Fig. 1: Flowchart for isolation of starch from tiger nut tuber: Source: Alabi *et al.*[16]

Modification of starch:

Oxidation of starch: Oxidation of native starches was carried out using the method adopted by Ganiyat *et al.*[17]. Starch slurry was prepared by adding 100 g of starch to 500 mL of distilled water in a 1 L reaction vessel. The pH of the slurry was adjusted to 9.5 with 2 M NaOH. Sodium hypochlorite (10 g),

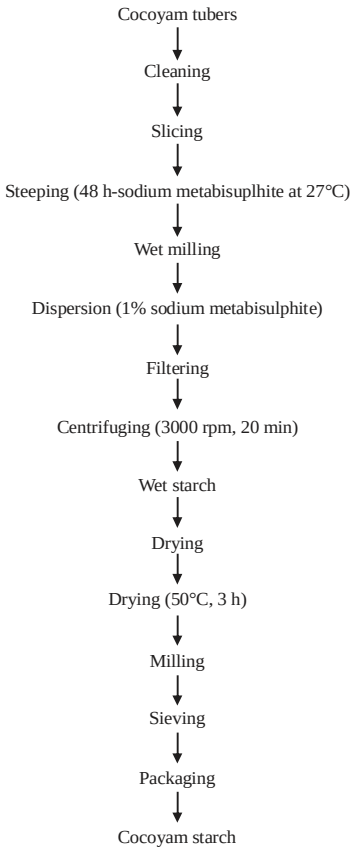

Fig. 2: Flowchart for isolation of starch from cocoyam: Source: Alabi *et al.*[16]

was added slowly added into the slurry over a period of 30 min while maintaining the pH range at 9.0-9.5, with constant stirring at 30±2°C. The reaction proceeded for 10 min after addition of NaOCl. After the reaction the pH of the slurry was adjusted to 7.0 with 1 M H_2SO_4 and the oxidized starch was filtered, washed four times with distilled water and air-dried at 30±2°C for 48 h.

Acetylation of starch: Acetylation of native starches was carried out using the method adopted by Ganiyat *et al.*[17]. Starch (100 g) was dispersed in 500 mL of distilled water and the mixture was stirred mechanically for 20 min. The pH of the slurry was adjusted to 8.0 using 1 M NaOH. Acetic anhydride (10.2 g) was added over a period of 1 h, while maintaining a pH range of 8.0-8.5. The reaction was allowed to proceed for 5 min after the addition of acetic anhydride. The pH was adjusted to 4.5 using 0.5 M HCl. It was filtered, washed four times with distilled water and air dried at 30±2°C for 48 h.

Pre-gelatinization of starch: Pre-gelatinization of native starches was carried out using the method adopted by Ganiyat *et al.*[17]. A 300 g of starch was suspended in 1 L of distilled water and heated to 80°C for 15 min with slow mixing. Pre-gelatinized starch was placed into stainless steel tray in form of thin film (1-2 mm) and dried in a convection oven at 40°C for 48 h, it was milled to pass through a 100 µm mesh screen and was stored at room temperature (30±2°C) in sealed glass jar.

Dual modification (Acetylation-oxidation): Dual acetylated-oxidized starch was produced using the method of Khan *et al.*[19]. The oxidized starch that was produced was further acetylated, which was done as per the above procedure mentioned above.

Determination of proximate composition of starch

Determination of starch moisture content: The moisture content both native and chemically modified starch was determined by the method described by AOAC[20]. About 5 g sample was weighed into a Petri dish of known weight. It was then dried in the oven at 105±1°C for 4 h. The samples were cooled in a dessicator and then weighed. The moisture content was calculated as follows:

$$\text{Moisture content (\%)} = \frac{AB}{C} \times 100 \quad (1)$$

Where:

A-B = Change in weight

C = Initial weight of the food before drying

Determination of protein content of starch: The protein content was determined using micro-Kjedhal method of AOAC[20] which involves wet digestion, distillation and titration. The protein content was determined by weighing 3 g of the sample into a boiling tube that contains 25 mL concentrated sulfuric acid and one catalyst tablet containing 5 g K_2SO_4, 0.15 g $CuSO_4$ and 0.15 g TiO_2. Tubes were heated at low temperature for digestion to occur. The digest was diluted with 100 mL distilled water, 10 mL of 40% NaOH and 5 mL $Na_2S_2O_3$, anti-bumping agent was added and then the sample was diluted with 10 mL of boric acid. The NH_4 content in the distillate was determined by titrating with 0.1 N standard HCl using a 25 mL burette. A blank was prepared without the sample. The protein value obtained was multiplied by a conversion factor and the result was expressed as the amount of crude protein.

Calculation:

$$\text{Crude protein} = \frac{\text{Actual titre value - Titre of the blank} \times 0.1\text{N HCl} \times 0.014 \times \text{C.f}}{\text{Weight of sample}} \times 100 \quad (2)$$

Where:

Cf = Conversion factor (6.5)

Determination of fat content of starch: Fat content was determined using the method of AOAC[20]. About 10 g of the sample wrapped in filter paper was weighed using a chemical balance. It was then placed in an extrusion thimble that was previously clean, dried in an oven and cooled in the dessicator before weighing. Then, about 25 mL of petroleum ether was measured into the flask and the fat content was extracted. After extraction, the solvent was evaporated in the oven. The flask and the content were cooled in a dessicator and weighed.

Calculation:

$$\text{Percentage of total fat content} = \frac{\text{Weight of fat extracted}}{\text{Weight of food sample}} \times 100 \quad (3)$$

Determination of crude fibre content: Crude fibre was determined using the method of Adebowale and Sanni[21]. About 5 g of each sample was weighed into a 500 mL

Erlenmeyer flask and 100 mL of TCA digestion reagent was added. It was then brought to boiling and reflux foe exactly 40 min counting from the start of boiling. The flask was removed from the heater, cooled a little and filtered through a 15.0 cm No. 4 Whatman paper. The residue was washed with hot water stirred once with a spatula and transferred to a porcelain dish. The sample was dried overnight 150°C. After drying, it was a transferred to dessicator and weighed as W_1. It was then burnt in a muffle furnace at 500°C for 6 h, allowed to cool and reweighed as W_2.

$$\text{Percentage crude fibre} = \frac{W_2 - W_1}{W_0} \times 100 \quad (4)$$

Where:

W_1 = Weight of crucible + fibre +ash
W_2 = Weight of crucible +ash
W_0 = Dry weight of food sample

Determination of ash content: Ash content was determined using the method of Adebowale and Sanni[21]. About 5 g of each sample was weighed into crucibles in duplicate and then the samples were incinerated in a muffle furnace at 550°C until a light grey ash was observed and a constant weight obtained. The samples were cooled in the dessicator to avoid absorption of moisture and weighed to obtain ash content.
Calculation:

$$\text{Ash (\%)} = \frac{A - B}{C} \times 100 \quad (5)$$

Where:

A = Weight of crucible with sample
B = Weight of crucible with ash
C = Weight of sample

Determination of carbohydrate content: The carbohydrate was calculated as weight by difference between 100 and the summation of other proximate parameters as Nitrogen Free Extract (NFE) percentage carbohydrate:

$$NFE = 100 - (M + P + F + A + C_f) \quad (6)$$

Where:

M = Moisture content
P = Protein content
F = Fat content
A = Ash content
C_f = Crude fibre

Determination of functional properties determination of starch

Determination of swelling and solubility properties: Swelling power and solubility profile of starch was determined according to the method of Adebowale and Sanni[21]. Starch sample (5 g) was weighed into a pre-weighed centrifuge tube and 20 mL of distilled water was added and thoroughly shaken on a vortex. It was then heated to temperatures of 50, 60, 70 and 80°C for 30 min in a water bath. The sample was centrifuged at 3000 rpm for 20 min. The supernatant was decanted carefully and residue was weighed for swelling power determination. The supernatant decanted was dried to a constant weight at 110°C in hot air oven. The residue obtained after drying the supernatant represents the amount of soluble starch solubilized in water.
Calculation:

$$\text{Swelling power} = \frac{\text{Weight of sediment}}{\text{Sample weight} - \text{Weight of soluble}} \quad (7)$$

$$\text{Solubility (\%)} = \frac{\text{Weight of soluble}}{\text{Weight of sample}} \quad (8)$$

Determination of water absorption capacity: The water absorption capacity of the starch was determined according to the method of Claver *et al.*[22]. Distilled water (10 mL) was added to 1 g of sample and the mixture was mixed thoroughly using a vortex mixer for 30 sec and centrifuge at 4000 rpm for 10 min. The mass of absorbed was expressed as g g^{-1} starch on a dry weight basis.

Determination of bulk density: Bulk density was determined according to the method of Musa *et al.*[23]. Starch (30 g) was weighed into a 25 mL measuring cylinder and the volume occupied was measured and recorded.
Calculation:

$$\text{Bulk density (g mL}^{-1}) = \frac{\text{Weight of samples}}{\text{Volume occupied by sample}} \quad (9)$$

Determination of starch pH: The pH was determined according to the method to the method adopted by Zeiba *et al.*[24]. A 20% w/v dispersion of the sample was taken into water for 5 min and the pH was determined using a pH meter.

Determination of oil absorption capacity: Oil Absorption Capacity (OAC) was determined using the method adopted by

Ganiyat *et al.*[17]. Starch sample was mixed with 20 mL sunflower oil and was allowed to stand at room temperature (30±2°C) for 30 min, then centrifuged for 30 min at 2000 rpm (527×g). The volume of decanted supernatant fluid was measured and the volume of oil retained/bound per g of sample calculated. Oil Absorption Capacity (OAC) was expressed as g of oil bound/100 g of starch.

Determination of gelatinization temperature: This was evaluated by the method of Attama *et al.*[25]. The starch sample (1 g) was put in a 20 mL beaker and 10 mL of distilled water was added. The dispersion was heated on a hot plate. The gelatinization temperature was read with a thermometer suspended in the starch slurry.

Determination of starch viscosity: Ten percent (10%) of the starch was suspended in distilled water and mechanically stirred for 2 h at room temperature. The Ostwald viscometer was used to measure the viscosity.

Determination of starch paste clarity: Paste clarity was determined using the modified method of Henry[26] using light transmittance of starch paste (%). The paste (1%) was produced when starch (0.05 g) was suspended in 5 mL of distilled water in screw cap tubes and then was placed in boiling water for 30 min. The tube was thoroughly shaken every 5 min. After at room for 5 min, the percentage transmittance at 650 nm was determined against a water blank in a spectronic 21 spectrophotometer.

Determination of colour dimension of starch: The colour characteristics of the starch samples was measured using a color measuring instrument (Model SN 3000421, Color TEC-PCM,USA) according to the method of Matthew and Ojo[27] and the values expressed on the L*, a*, b*. Where L* represent lightness from 0 (black) to 100 (white); a* and b* represent redness (+a) to greenness (-a) and yellowness (+b) to blueness (-b), respectively. Three grams (3 g) of the starch sample was put in clean paper and the color meter was placed on the sample by allowing the sensor to touch the sample and the reading was taken directly.

RESULTS AND DISCUSSION

The result in Table 1 and 2 show the proximate composition of native and modified cocoyam starch and native and modified tigernut starch respectively. There were some significant differences in the values obtained at $p \leq 0.05$. The moisture content which is a factor of storability ranges from 7.43 to 11.34%, with dual modified cocoyam starch (DS) having the lowest and pregelatinized cocoyam starch (PS) having the highest moisture content. The protein content ranges from 0.68 to 0.51%, with pregelatinized cocoyam starch (PS) having the lowest and DS is having the highest protein content. Dual modified cocoyam starch (DS) tends to have the highest crude fat (0.19%) and pregelatinized cocoyam starch (PS) (0.07%) having the least value for crude fat. The ash content ranges from between 1.93 to 2.39% in which oxidized tiger nut starch (OS) had the highest value and dual modified cocoyam starch (DS) having the least. The carbohydrate content which is a source of energy ranges from 89.76 to 87.56%, where sample OS had the lowest value and sample DS had the highest value.

The result on Table 2 shows the proximate composition of native and modified tiger nut starch. The moisture content which determine the shelf stability of the starch ranges from 6.58 to 15.43%, with sample NS having the lowest and sample AS having the highest moisture content. The protein content of the starch was very low, with sample PS (0.27%) having the least value and sample DS having the highest value of protein. The crude fat ranges from between 2.87 to 28.45% with sample PS having the least fat than sample DS having the highest value of fat. The crude fibre ranges from 0.13 to 0.28%, where sample NS had the lowest crude fibre compared to sample DS that had the highest. The total ash content of sample NS was the lowest (0.09%) compared to the modified starch while sample DS has the highest value of total ash. The carbohydrate content was a good source of calories which ranges from 52.50 to 82.26%, in which sample DS had the

Table 1: Proximate composition of cocoyam starch as influenced by modification

Sample	Moisture	Protein	Crude fat	Crude fibre	Total ash	CHO
	(%)					
NS	7.52±0.03^{b}	0.63±0.02^{c}	0.13±0.01^{c}	0.05±0.01^{b}	2.01±0.01^{b}	89.67±0.50^{d}
OS	9.33±0.01^{d}	0.54±0.01^{b}	0.09±0.01^{b}	0.09±0.01^{d}	2.39±0.01^{e}	87.56±0.02^{b}
AS	9.17±0.01^{c}	0.65±0.01^{d}	0.15±0.06^{d}	0.06±0.01^{c}	2.09±0.01^{c}	87.88±0.03^{c}
PS	11.34±0.02^{e}	0.51±0.01^{a}	0.07±0.00^{a}	0.07±0.00^{c}	2.31±0.17^{d}	85.70±0.03^{a}
DS	7.43±0.01^{a}	0.68±0.00^{e}	0.19±0.01^{e}	0.02±0.01^{a}	1.93±0.12^{a}	89.76±0.03^{d}

Values are means of triplicate and standard deviation, different letters within column are significantly ($p<0.05$) different from each other, NS: Native Starch, OS: Oxidized Starch, AS: Acetylated Starch, PS: Pregelatinized Starch, DS: Dual modified Starch

Table 2: Proximate composition of tiger nut starch as influenced by modification

Sample	Moisture	Protein	Crude fat	Crude fibre	Total ash	CHO
	(%)					
NS	6.58±0.01[a]	0.34±0.01[a]	13.76±0.02[c]	0.13±0.01[a]	0.09±0.01[a]	78.29±0.05[c]
OS	7.75±0.03[b]	0.30±0.01[b]	9.24±0.01[b]	0.25±0.01[d]	2.23±0.02[d]	80.23±0.05[d]
AS	15.43±0.02[e]	0.43±0.12[d]	20.77±0.17[d]	0.18±0.00[b]	1.38±0.01[b]	61.81±0.16[b]
PS	8.57±0.06[c]	0.27±0.01[a]	2.87±0.12[a]	0.20±0.01[c]	1.85±0.01[c]	86.26±0.06[e]
DS	13.38±0.01[d]	0.49±0.01[e]	28.45±0.12[e]	0.28±0.01[e]	4.90±0.01[e]	52.50±0.01[a]

Values are means of triplicate and standard deviation, different letters within column are significantly ($p<0.05$) different from each other, NS: Native Starch, OS: Oxidized Starch, AS: Acetylated Starch, PS: Pregelatinized Starch, DS: Dual modified Starch

lowest value and sample PS had the highest carbohydrate content. The dual modified cocoyam starch and the native tiger nut starch had the lowest moisture content value of 7.42 and 6.58%, respectively while pregelatinized cocoyam starch and acetylated tiger nut had the highest moisture content value of 11.34 and 15.43% which is higher than moisture content of native and modified 'kponan' yam as reported by Kone *et al.*[28] also the moisture content of native cocoyam starch (7.52%) was lower to that reported by Tijani *et al.*[5], who reported native cocoyam starch to have 9.57% and higher moisture content for acetylated cocoyam starch to be 11.73% which is lower to the acetylated cocoyam result above having 9.17%. Therefore, sample DS for cocoyam starch and sample NS for tigernut starch will be more shelf stable than others because the lower the moisture content, the higher the shelf stability and reduces microbial activities of the food sample. The moisture content of a powder plays a significant role in the flow and other mechanical properties of the food. However, this may depend largely on the method, extent of drying and the humidity in the surrounding atmosphere. From the above result, there was significant decrease in moisture content of sample DS cocoyam and sample NS of tiger nut, which indicate that the samples might have better storage stability.

The protein content of dual modified cocoyam starch and dual tigernut starch had the highest of 0.68 and 0.49%, respectively and pregelatinized cocoyam starch and pregelatinized tigernut starch had the least value of 0.51 and 0.27%, respectively, lower than pregelatinized banana and plantain starch reported by Ganiyat *et al.*[17]. Kone *et al.*[28] reported native, oxidized and acetylated 'Kponan' yam cultivar starch to have 0.18, 0.14, 0.15% which was lower than the protein content of both the native, oxidized and acetylated starch of both the tiger nut and cocoyam. This may be due to the agronomy process, the type of fertilizer used and the amount of nitrogen fixation in the soil which tends to increase the protein content of the cocoyam starch. Ojinnaka *et al.*[29] reported that cocoyam has more crude protein than roots and other tubers and its starch is highly digestible because of the small of the starch granules. Dual modified cocoyam starch and tiger nut starch had the highest crude fat content of 0.19 and 28.45%, respectively and oxidized cocoyam starch and pregelatinized tigernut starch had the least value of 0.09 and 2.87%, respectively. The result generated for both native starch and modified tigernut starch in this report is higher than that reported by Ganiyat *et al.*[17] for native and modified banana and plantain starch. This was because tiger nut is a good source of edible fat and starch and oil are major macronutrients in the tiger nut tuber. High starch content of this plant provides unique functional properties[12], cold storage stabilities and preserves organoleptic properties of foods[30]. Dietary fats that provide Essential Fatty Acids (EFA) have been shown to enhance the taste and acceptability of food, slow gastric emptying and intestinal motility, thereby prolonging satiety and facilitate the absorption of lipid-soluble vitamins[31]. Oxidized cocoyam starch and dual modified tigernut starch had the highest crude fibre value of 0.09 and 0.28%, respectively and dual modified cocoyam starch and native tigernut starch had the least value of 0.02 and 0.13%, respectively. Tiger nut was reported to be high in dietary fibre content which could be effective in the treatment and prevention of many diseases including colon cancer, coronary heart diseases, obesity, diabetics and gastro intestinal disorder[32]. Oxidized cocoyam starch and dual modified tigernut starch had the highest ash value of 2.39 and 4.90%, respectively and dual modified cocoyam starch and native tigernut starch had the least value of 1.93 and 0.09%, respectively. The increase in ash content could make a product a good source of minerals as observed by Borchani *et al.*[33]. Dual modified cocoyam starch and pregelatinized tigernut starch had the highest value of 89.76 and 86.26%, respectively while pregelatinized cocoyam starch and dual modified tigernut starch had the least value of 85.70 and 52.50%, respectively. Therefore, cocoyam and tiger nut starch are both good source of carbohydrate which is an energy given food. In addition, a lower content of protein, fat and ash indicate purity of a starch which may be directly linked to a method of starch isolation[17].

Table 3: Functional properties of cocoyam starch influenced by modification

Sample	Swelling (%)	Solubility (%)	WAC (%)	BD (g mL^{-1})	pH	OAC (%)	Gel. Temp. (°C)	Viscosity (CP)	Paste clarity (%)
NS	4.66±0.88^{a}	7.33±0.58^{a}	1.40±0.17^{b}	0.72±0.01^{a}	5.48±0.09^{a}	0.40±0.17^{a}	72.67±2.31^{a}	1986.67±1.12^{c}	21.62±0.03^{d}
OS	8.74±0.10^{b}	8.00±0.00^{a}	1.07±0.12^{a}	0.68±0.12^{a}	5.85±0.03^{b}	0.43±0.12^{a}	71.67±1.53^{a}	1221.00±1.73^{a}	18.50±0.00^{a}
AS	6.14±0.06^{a}	7.67±1.12^{a}	1.00±0.00^{a}	0.66±0.01^{a}	5.46±0.03^{a}	0.53±0.06^{a}	75.67±0.58^{b}	127.33±2.31^{b}	18.93±0.03^{b}
PS	11.76±1.82^{c}	10.33±1.15^{b}	1.07±0.06^{a}	0.62±0.01^{a}	5.75±0.03^{b}	0.30±0.17^{a}	76.00±0.00^{b}	2188.33±0.56^{e}	23.33±0.29^{e}
DS	4.78±0.06^{a}	8.00±1.73^{a}	1.33±0.15^{b}	2.69±3.45^{a}	5.73±0.13^{b}	1.07±0.12^{b}	80.33±1.53^{c}	2061.67±1.12^{d}	20.16±0.01^{c}

Values are means of triplicate and standard deviation, different letters within column are significantly ($p<0.05$) different from each other, NS: Native starch, OS: Oxidized starch, AS: Acetylated starch, PS: Pregelatinized Starch, DS: Dual modified Starch

Table 4: Functional properties of tiger nut starch as influenced by modification

Sample	Swelling (%)	Solubility (%)	WAC (%)	BD (g mL^{-1})	pH	OAC (%)	Gel. Temp. (°C)	Viscosity (Cp)	Paste clarity (%)
NS	8.02±1.17^{b}	10.50±0.71^{ab}	1.50±0.00^{a}	0.59±0.01^{b}	5.56±0.06^{ab}	0.20±0.00^{a}	62.50±3.54^{a}	873.00±61.41^{a}	9.00±0.00^{a}
OS	6.08±1.59^{ab}	9.00±2.83^{ab}	1.55±0.00^{a}	0.54±0.06^{ab}	5.79±0.26^{ab}	0.35±0.21^{a}	69.00±5.66^{a}	940.50±96.87^{ab}	8.75±0.35^{a}
AS	5.02±0.00^{a}	7.50±0.71^{a}	2.00±0.07^{a}	0.51±0.01^{a}	5.94±0.05^{b}	0.55±0.07^{a}	67.50±3.54^{a}	1011.00±71.83^{b}	8.50±0.00^{a}
PS	4.36±0.50^{a}	12.50±0.71^{b}	1.00±0.00^{a}	0.53±0.01^{ab}	5.66±0.01^{ab}	0.35±0.21^{a}	80.50±0.71^{b}	1065.50±70.71^{b}	16.31±0.21^{b}
DS	4.32±0.53^{a}	10.50±2.12^{ab}	1.75±1.06^{a}	0.52±0.01^{ab}	5.50±0.24^{a}	0.75±0.35^{a}	80.50±0.71^{b}	1027.00±53.74^{b}	15.26±1.51^{b}

Values are means of triplicate and standard deviation, different letters within column are significantly ($p<0.05$) different from each other, NS: Native Starch, OS: Oxidized Starch, AS: Acetylated Starch, PS: Pregelatinized Starch, DS: Dual modified Starch

Functional properties of modified and unmodified starch: The result on Table 3 and 4 show the functional properties of both cocoyam and tigernut, respectively. The tables revealed that there are significant differences ($p<0.05$) among the functional properties of each modified cocoyam starch. The swelling capacity ranged from 4.66 to 11.76% with sample NS having the least value and sample PS having the highest value. The swelling capacity varies of sample DS varies significantly with sample NS. The solubility index ranged from 7.33 to 10.33% Sample NS having the least value and sample PS having the highest value while sample OS and DS were not significantly different from each other. The Water Holding Capacity (WAC) ranged from 1.00 to 1.40% with sample AS having the lowest value and sample NS having the highest value while sample OS and PS were not significantly different from each other. The bulk density ranged from 0.62 to 2.69 g mL^{-1} with sample PS having the lowest value and sample DS having the highest value. There is significant difference of ($p\leq0.02$) between sample OS and sample AS. The pH which measured the acidity of the sample ranged from 5.46 to 5.85 where sample AS had the lowest value and sample OS had the highest value. There were significant differences ($p<0.05$) across the samples. The Oil Absorption Capacity (OAC) ranged from 0.03 to 1.07% where sample PS had the lowest value and sample DS had the highest value. The gelatinization temperature ranged from 71 to 80°C where sample OS had the least value and sample DS had the highest value. The viscosity ranged from 127CP to 2188CP with sample AS having the least value and sample PS having the highest value. Also, the paste clarity of the starch varies from 18.50 to 23.33% where sample OS had the lowest value and sample PG having the highest value. The result in Table 4 shows the functional properties of native and modified tiger nut starch. The swelling capacity ranged from 4.32 to 8.02% with sample DS having the least value and sample NS having the highest value. The solubility index ranges from 7.50 to12.50% with sample AS having the least value and sample PS having the highest value while sample NS and DS had no significant difference. The Water Absorption Capacity (WAC) ranged from 1.00 to 2.00% where sample PS had the lowest value and sample AS had the highest value, the WAC varied significantly across samples. The bulk density ranged from 0.51 to 0.59 g mL^{-1} where sample AS had the least value and sample NS had the highest value and there is slight significant difference between across the sample for bulk density. The pH value which determines the acidity ranges from 5.50 to 5.94 where sample DMS had the least value and sample AS having the highest value. The Oil Absorption Capacity (OAC) ranges from 0.20 and 0.75% where sample NS had the lowest value and sample DS had the highest value while sample OS and PS had the same value of OAC. The gelatinization temperature ranges from 62.50 to 80.50°C where sample NS had the least value and sample PG and DMS had the highest value. The viscosity ranges from 873.00 to 1065 CP where sample NS had the least value and sample PG had the highest value. The paste clarity ranges from 8.50 to 16.31% where sample AS had the lowest value and sample PG had the highest.

The swelling power and solubility index were usually used to assess the extent of interaction between starch chains within the amorphous and the crystalline domains of the starch granule[5]. Swelling capacity was the ability of starch granules to hydrate and increase in size under excess water condition also starch solubility was considered as an indicator of the degree of dispersion of molecule of starch granules after

cooking[34]. The swelling and solubility index of cocoyam starches ranges from 4.66 to 11.76% and 7.33 to 10.33%, respectively, where native cocoyam had the lowest value and pregelatinized cocoyam had the highest value. Tiger nut starches also ranges from 4.32 to 8.02% and 7.50 to 12.50%, respectively higher than that reported for native and modified banana and plantain starch by Ganiyat *et al.*[17], where dual modified tiger nut starch had the lowest swelling capacity and native tiger nut starch had the highest value and acetylated tiger nut starch had the lowest solubility index and pregelatinized tiger nut starch had the highest solubility index. The increase in the swelling and solubility index may be as a result of granule size and amylose content of the cocoyam starch[5]. Indeed starch with large granules swells rapidly when heated in water and water molecule are bonded to the free hydroxyl groups of amylose and amylopectin by hydrogen bond. Previous studies have indicated that greater swelling capacity is an indication of weaker binding force in the starch granules[16]. The water absorption capacity of cocoyam starch ranged between 1.00 and 1.40% where acetylated cocoyam starch had the least value and native cocoyam starch had the highest value lower than the value obtained for native water yam starch and acetylated starch having 59.4 and 119.1%, respectively, by Matthew and Ojo[27], while tiger nut ranges from 1.00 to 2.00% where pre-gelatinization had the least and acetylation had the highest value. The acetylated process generally led to the increased in the water absorption capacity of modified starches[17]. The ability of starches to absorb water is a function of hydrophilic groups (-OH,-COOH, inter-glucose oxygen atoms) available for binding of water, presence of phosphate-esters or proteins, crystallinity and botanical source. High WAC was an indication of a loose association of starch polymers in the granules of the starch and low WAC results from the compactness of molecular structure of starch[16]. Studies have shown that the microbial activities of food product with low WAC can be reduced; hence, the shelf life of the product can be extended. It dictates the water holding power of the starch when used in soups as thickeners. Bulk density is the ratio of the mass per unit volume of a substance. It is an indication of the porosity of a product which influences packaging. The bulk density of native and modified cocoyam starch ranged between 0.62 and 0.72 g cm^{-3} although they are not significantly different ($p<0.05$) from each other while tigernut ranged between 0.51 and 0.59 g cm^{-3}. The bulk density obtained in this report for cocoyam starch was higher than 0.62 g mL^{-1} reported for tiger nut flour and 0.71 g cm^{-3} for wheat flour[34,35]. The bulk density implies that less quantity of food samples would be packaged in constant volume thereby ensuring an economical packaging. pH is an important property in starch industrial applications, being used generally to indicate the acidity and alkalinity of a liquid media. The pH of cocoyam starch ranges from 5.46 to 5.85 with acetylated cocoyam having the lowest value and oxidized having the highest value and tiger nut starch ranges from 5.50 to 5.94 with dual modified tiger nut starch having the least value and acetylated tiger nut starch having the highest value. The pH was closer to that reported by Zeiba *et al.*[24] having tiger nut starch as 5.60±0.1 and maize starch 5.92±0.2. It was comparable with the previous pH reported for tuber starches and within the pH range of 3-9 obtained for most starches used in pharmaceutical, cosmetics and food industries[24]. The oil absorption capacity (OAC) of native and modified cocoyam starch ranged between 0.30 and 1.07% for pregelatinized starch been the lowest and dual modified been the highest and the OAC of tiger nut starch ranges from 0.20 to 0.75% where native tiger nut starch had the lowest value and dual modified starch having the highest value. The high value of oil absorption was an indication that tiger nut flour will be a useful material in snack production[16]. Confectionaries and food ingredients such as thickeners will require starch with higher WAC while products such as batter for frying and mayonnaise will require starch with appropriate OAC[36]. The low OAC suggest that the starches are fairly useful in structural interaction in food such as flavor retention, especially food where fat absorption is desired. The gelatinization temperature ranged from 71.67 to 80°C for cocoyam starch where oxidized starch had the least value and dual modified had the highest value and tiger nut starch ranged from 62.50 to 80.50°C where native tigernut starch had the least value and pre-gelatinization and dual had the highest value. The result obtained was quite similar to that reported by Zeiba *et al.*[24] on tiger nut and maize starch having 66 and 73°C, respectively. The starch was observed to have a gelatinization temperature of 66°C which falls within the gelatinization commonly observed for starches. The paste clarity varied between 18.50 to 23.33% for cocoyam starch where oxidized cocoyam starch had the least value and pregelatinized had the highest value and for tiger nut starch it varied between 8.50 to 16.31% where acetylated had the least value and pre-gelatinization had the highest value. Indeed, high paste clarity observed for starch signifies that the starch granules for these samples are fragile during pasting and remnant of granules are absent from the paste[28]. On the other hand, the low paste clarity found in oxidized cocoyam starch and acetylated tigernut may be explained by the presence of molecules less susceptible to retrogradation.

Besides, Kone *et al.*[28] suggested that more opaque paste gave low paste clarity (% Transmittance). Indeed, an increase in opaque paste during storage could be attributed to factors like leached amylose and amylopectin chains, granular remnants, granular swelling, amylose and amylopectin chain length. Also, the highest paste clarity obtained in this study may find application in food industry while the lower paste clarity in this study which is required for low transparence may also find application in gravies and thickened food[5]. The viscosity of the native and modified cocoyam starch ranged from 127 to 2188 CP with acetylated starch having the least value and pre-gelatinization starch having the highest value while of tiger starch ranged from 873.00 to 1065 CP where native starch had the least value and pregelatinized starch had the highest value.

Colour analysis of native and modified starches: Colour is one of the most important attributes of food materials, as it influences consumer acceptability hence colour was also used in process control. Colour may be controlled by the degree of colour formation because the brown pigments increase as heating or browning and caramelization reactions progress. The lightness index of cocoyam starch ranged from 97.78 to 108.34 having the pregelatinized cocoyam starch as the highest and the oxidized starch as the lowest value. The pregelatinized starch tends to be the lightest (108.35) compared to native starch with L*-value 0f 100.06 which was as well higher than the L* value of native cocoyam starch (46.72) reported by Alabi *et al.*[16] and the oxidized starch, acetylated starch and dual modified starch with 97.78, 98.14 and 98.63, respectively are less lighter compare to the native and pregelatinized starch. The hue angle (H*) gave negative value for both native and modified starch, indicating that there was no characteristic colour in the sample other than white colour. The light intensity value ranged from 86.25 to 96.25 where acetylated had the lowest value and pregelatinized starch had the highest value. The chroma value ranges from 11.86 to 13.11 where acetylated starch had the lowest value and pregelatinized starch had the highest value. The chroma increased in the pregelatinized cocoyam against the native starch. The chroma has been reported to be a measure of colour purity in a material[27]. A low value of chroma and a high value of lightness are desired to meet consumer's preference[16]. Therefore, the native starch and pregelatinized are desirable to meet consumer's preference. The '±a*' is regarded as the measure of the degree of redness or greenness while '±b*' represent the degree of yellowness or blueness in a material. The '±a*' are negative thus indicating the absence of red or green colour. However, these factors may not be useful for describing the colour characteristics of these starches although their popular use lies in the visual assessment in fruit ripening[27]. The hue angle (H*) gave negative value for both native and modified starch, indicating that there was no characteristic colour in the sample other than white colour. The result in Table 5 and 6 shows the colour analysis of cocoyam starch and tiger nut native and modified starches.

The lightness L*-value ranged from 59.17 to 108.34 where native starch had the lowest value and pregelatinized starch had the highest value. In this report, the L*-value of native starch (59.17) was higher than that reported by Alabi *et al.*[16] having native tigernut L*value as (38.5). Modification increases the lightness of the other starches than the native starch, making pregelatinized starch the lightest of them all. There

Table 5: Colour analysis of cocoyam starch as influenced by modification

Samples	L*	a*	b*	H*	C*	^E
NS	100.06±0.44^b	-5.83±0.05^a	10.97±0.05^a	-62.00	12.42	88.23±0.44^b
OS	97.78±0.15^a	-6.16±0.01^b	10.63±0.01^a	-59.97	12.29	89.95±0.15^c
AS	98.14±0.31^a	-6.13±0.02^b	10.15±0.03^a	-58.93	11.86	86.25±0.31^a
PS	108.34±0.25^c	-6.26±0.01^c	11.52±0.02^b	-61.48	13.11	96.52±0.25^d
DS	98.63±0.31^a	-6.22±0.02^c	10.20±0.04^a	-58.63	11.95	86.74±0.31^a

Values are means of triplicate and standard deviation, different letters within column are significantly ($p<0.05$) different from each other, NS: Native starch, OS: Oxidized Starch, AS: Acetylated Starch, PS: Pregelatinized Starch, DS: Dual modified starch

Table 6: Colour analysis of tiger nut starch as influenced by modification

Sample	L*	a*	b*	H*	C*	^E
NS	59.17±1.01^a	-0.84±0.01^a	13.46±0.24^a	-85.47	13.49	48.28±1.03^b
OS	97.78±0.15^b	-1.17±0.02^b	12.71±0.05^b	-84.74	12.76	47.63±0.20^b
AS	98.14±0.31^b	-1.27±0.02^b	13.87±0.45^a	-58.87	13.93	51.56±2.04^c
PS	108.34±0.25^c	-1.04±0.01^b	12.40±0.04^b	-61.48	12.44	44.39±0.22^a
DS	98.63±0.31^d	-1.67±0.01^c	13.74±0.00^a	-58.62	13.84	54.22±0.02^d

Values are means of triplicate and standard deviation, different letters within column are significantly ($p<0.05$) different from each other, NS: Native Starch, OS: Oxidized Starch, AS: Acetylated Starch, PS: Pregelatinized Starch, DS: Dual modified Starch

was no significant difference between oxidized starch and acetylated starch at ($p<0.05$). The chroma value ranged from 12.44 to 13.93 where pregelatinized starch had the lowest value and native starch had the highest value. The chroma has been reported to be a measure of colour purity in a material[27]. A low value of chroma and a high value of lightness are desired to meet consumer's preference[16]. In this report, it shows that pregelatinized starch possessed the highest L* value (108.34) and the lowest chrome value (12.44), therefore it was desirable to meet consumer's preference. The value for chroma (C*) ranges from 44.39 to 54.22, where pregelatinized starch have the lowest value and dual modified starch have the highest value. The '±a*' is regarded as the measure of the degree of redness or greenness while '±b*' represent the degree of yellowness or blueness in a material. The '±a*' are negative thus indicating the absence of red or green colour. However, these factors may not be useful for describing the colour characteristics of these starches although their popular use lies in the visual assessment in fruit ripening[27].

CONCLUSION

It was evident from the results of this study that modification processes were effective in altering and improving the characteristics of cocoyam and tiger nut starches. Dual modification of cocoyam starch tends to improve chemical composition and some functional properties except for water absorption capacity. Dual modified tigernut starch had highest value of protein, crude fat, fibre, which can be used for product that required any of the nutrients, therefore may be used as alternative food grade starch additive for industrial purposes, thereby encouraging import substitution. Therefore, modified cocoyam and tiger nut starch should be intensify in order to find new food applications and to determine their potential as substitute to other starches needed in food industries.

Research on cocoyam and tigernut starch modification should be intensified in order to find new food applications and to determine their potentials substitute to other starches needed in food industry. Government should also encourage the substitution of some part of wheat flour with modified cocoyam and tigernut starch in the production of baked products, this will help minimize the rate of postharvest losses and encourage cocoyam and tigernut cultivation.

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