



Structural and Physicochemical Characteristics of Six Edible Tubers Cultivated in Benin City, Nigeria

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ABSTRACT

Quality tubers are required for industrial processes and their physicochemical contents vary from one region to another. This work determined the physicochemical differences of starch in six species of edible tubers (*Dioscorea alata*, *Dioscorea bulbifera*, *Dioscorea cayenensis*, *Dioscorea rotunda*, *Manihot esculenta* and *Colocasia esculenta*) harvested in Benin City, Nigeria from September-November, 2021, as potential sources of dietary substances and for industrial use. Methods for analysis were as described in the Official Methods of the Association of Analytical Chemists. The texture solution of *Dioscorea cayenensis* and *Colocasia esculenta* were opaque while the others were transparent and clear. Ash content were *Dioscorea cayenensis* ($0.23 \pm 0.02\%$), *Colocasia esculenta* ($0.19 \pm 0.01\%$), and *Dioscorea alata* ($0.13 \pm 0.02\%$). The moisture content was *Dioscorea alata* ($10.0 \pm 2.0\%$) while *Dioscorea cayenensis* ($5.00 \pm 0.5\%$) the least. Crude protein was *Dioscorea cayenensis* (0.21 ± 0.05) and least in *Dioscorea alata* ($0.07 \pm 0.02\%$). Lipid was *Dioscorea alata* ($0.22 \pm 0.03\%$) and least in *Dioscorea cayenensis* ($0.12 \pm 0.02\%$). Phosphorous was highest in *Dioscorea cayenensis* ($0.025 \pm 0.05\%$) and least in *Manihot esculenta* ($0.008 \pm 0.04\%$). Amylose values were *Dioscorea bulbifera* ($21.8 \pm 4.37\%$), *Dioscorea Dcayenensis* ($30.9 \pm 2.26\%$), *Dioscorea rotunda* ($29.8 \pm 3.41\%$), *Dioscorea alata* ($24.6 \pm 1.04\%$), *Manihot esculenta* ($20.5 \pm 2.50\%$), and *Colocasia esculenta* ($20.0 \pm 2.05\%$). The viscosity was *Colocasia esculenta* (0.800 ± 0.04) and least in *Dioscorea bulbifera* (0.200 ± 0.01). The density (0.99) was approximately the same. The pH was acidic except for *Dioscorea alata* (7.00 ± 1.0). The water binding capacity (WBC) was highest in *Dioscorea cayenensis* (1.15 ± 0.09) and least in *Dioscorea rotunda* (0.90 ± 0.25). The characteristics of these edible starches would enhance their industrial uses as well as improve their nutritional values to man.

Keywords: Dioscorea, Edible tubers, Proximate-analysis, Colocasia, Physicochemical analysis

Introduction

Tropical countries have great variety of roots and tubers, and these plants basically contain 70-80% water, 16-24% starch and quantities lower than 4% proteins and lipids.¹ They have many uses including medicinal.²⁻⁵ Structural and physicochemical characteristics of the contents of some plants such as amylose, branch chain length distribution of amylopectin, phosphate monoester, phospholipid contents affect their functional properties.⁶⁻⁷ Starches that contain amylopectin molecules with large proportion of long chains display higher gelatinization temperature.⁸⁻⁹ Phosphate monoester groups on the amylopectin decrease the gelatinization temperature of starches.¹⁰ The granular shape and size affect the functional properties of starches and may influence industrial application.¹¹ Starch is one of the most important natural organic compounds abundant in the roots or fruits of plants and its importance as a food and in industrial product has made it one of the most important plant products.¹² It can be obtained cheaply and in large amounts. Hence, it is flexible in application and can satisfy

demands in many processing and manufacturing ventures.¹³ Starch granules in higher plants are mostly composed of the polysaccharides amylose and amylopectin. Amylose is a predominantly linear polymer that contains 99% α (1-4) and 1.0% α (1-6) linkages. Amylopectin contains 95% α (1-4) and 5.0% α (1-6) linkages.¹⁴ Starch is the main It has the potential to be used to bind, expand, densify, clarify or opacify, absorb or block moisture. It has the ability to stabilize emulsions and exhibit a variety of textures, including stringy, silky, pulpy, soft, and crisp coatings.¹⁴ Starch granules from different botanical sources also vary in size, shape and content of amylose and amylopectin and all these affect their chemical and physical characteristics. Other applications include the creation of diverse surface textures including stringy, silky, or spongy surfaces and the stabilization of emulsions.¹⁵⁻¹⁷ Mucilage from some tubers and other roots has been shown to have both angiotensin activating inhibitory enzymes as well as anti-oxidative actions, however, some edible tubers do not contain gluten, and this of course is a determinant factor in sourcing for edible carbohydrates. Therefore, Celiac disease (CD) and related allergic reactions Yam isolation is critical for a variety of industrial applications. Along with other edible tubers, yam is commonly used as flour, starch, in industries for quality bread, biscuits and a host of pastries and even thickeners in soaps.¹⁷ The aim of this study was to extract starches from different tubers and investigate the properties of these starches and compare some structural and physicochemical characteristics of the starches isolated in six different species of edible tubers: *Dioscorea alata* (water yam), *Dioscorea bulbifera* (potato), *Dioscorea cayenensis* (yellow yam), *Dioscorea rotunda* (white yam), *Manihot esculenta* (cassava), and *Colocasia esculenta* (cocoyam) with the aim of improving the utilization of these starches in the food industry.

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Materials and Methods

Six edible tubers were studied in Benin City, Nigeria between September and November, 2021. They were identified at the Faculty of Agriculture, University of Benin, Benin City and following voucher numbers were given to them: *Dioscorea rotunda*, *Dioscorea cayenensis*, *Dioscorea alata*, *Manihot esculenta*, *Dioscorea bulbifera* and *Colocasia esculenta* UBH-D329, UBH-D605, UBH-D604, UBH-DM372, UBH-1433 and UBH-X603 respectively. The tubers were peeled, sliced and grounded with distilled water for 3 min with high speed blender. In the isolation processes, the starch was washed away by spraying water from a wash bottle onto the residue. The grinding and filtration of the residues were done thrice avoiding vigorous squeezing of the cheese cloth to improve the recovery of starch. The filtration was a milky filtrate that is rich in starch but contains other plant components such as mucilage, protein and fine fiber particles that can be separated out by centrifuge. Centrifugation was used to recover the precipitate from the resultant slurry after it had been allowed to stand overnight. After that, it was cleaned with ethanol and water before being dried at 380 degrees Celsius in an air circulation oven. For yam starch after the first centrifugation the mucilaginous layer was removed and the sediment was washed several times by suspension in distilled water until it appeared to be free of non-starch materials. The final precipitate was then washed with ethanol and dried¹⁸. Pure starch was then dried in the oven between temperatures of 30°C-40°C, and was hand ground to powder using mortar and pestle. The starch was sieved, weighed, labeled and stored in tightly closed container under dry condition.

Determination of moisture content

Measuring the water content of a substance Drying 3 g samples of starch in an air oven at 105 C for 24 hours gave us the moisture content. After thirty minutes in the desiccator, the samples were weighed and re-baked in the oven for another thirty minutes to maintain a steady weight. The moisture content (MC) of sample was expressed as a percentage.¹⁸

$$\text{Moisture content} = \frac{\text{Loss in weight}}{\text{Weight of sample}} \times \frac{100}{1}$$

Determination of the density

Pycnometer method was used to determine the density of the starches. A thoroughly clean and dried pycnometer was weighed and filled with starch solution and the weight was recorded. For this experiment, two grams of starch samples were placed in a beaker with 20 mL of distilled water, agitated for 5 minutes, and then permitted to settle for 10 minutes.¹⁸

Determination of pH

The pH of the water phase was measured using a calibrated pH meter

Determination of Water binding capacity (WBC)

Water binding capacity (WBC) was determined according to the method previously described¹⁹ with a few modifications. An aqueous suspension was made by dissolving 1g of starch in 20 ml of water. After 1 h of agitation, the suspension was centrifuged for 10 minutes. Water was decanted from the wet starch, drained for 10 min and weighed.

Determination of ash content

The ash content was determined in duplicate sets of crucibles of known weight containing dried starch samples. One gram was heated to 500 degrees Celsius in a muffle furnace for three hours before cooling in a desiccator and being weighed. Ash content was determined by weight difference and expressed as a percentage.¹⁸

$$\% \text{ Ash} = \frac{\text{Ash weight}}{\text{weight of sample used}} \times \frac{100}{1}$$

Determination of viscosity and swelling power of starch

The viscosity was determined by measuring the time of flow (seconds) through a tube. The time of flow through the capillary was taken as the

viscosity index since the study was of comparative purpose. The swelling power of starch was determined. Centrifuge tubes were filled with 2 grams of starch and 5 ml of distilled water and weighed. Tubes were then placed in a water bath and heated at 40, 50, 60, 70, 80 or 90°C for 30 min after equilibration of the sample temperature for 1 min with repeated shaking. For 15 minutes, the heated samples were placed in a cold water bath to rapidly cool them down to room temperature and then centrifuged at 3000 g for 15 min. The supernatant was removed carefully and the swelling power determined as the ratio of weight of starch sediment to original weight of starch before heating.

Determination of protein

Protein concentrations were measured using the Kjeldahl technique. One gram of dried sample was weighed into digestion flask, then 25ml of concentrated H₂SO₄ plus 1g of K₂SO₄ and CuSO₄. The flask was swirled in order to mix the content and then place on heater for 2-3 hr. to digest. The digestion was performed in marked distillation apparatus. In the distillation tube, ten milliliters of digest were added. The same method was used to add 30 ml of distilled water and 4 percent NaOH. Distillation continued for 10-15mins and NH₃ produced was collected as NH₄OH in a conical flask containing 5ml of mixed boric acid solution, with 3 drops of mixed indicator. The distillate was then titrated against standard 0.1N HCL solution. Percentage protein was calculated using the following formular.²⁰⁻²¹

$$\text{Crude protein} = 6.25 \times \% \text{ N}$$

$$\% \text{ N} = \frac{S \times M \times 0.014 \times V \times 100}{\text{Wt of sample used} \times V}$$

Where;

S= Sample titration reading

M= Molarity of acid

V1=volume of sample after digestion =100ml

V2=Volume of aliquot

0.014 = milli equivalent weight of nitrogen.

Weight of sample use =1.00g

Lipid extraction

Petroleum ether was used to reflux 2 grams of sample on a fat free filter paper. The Soxhlet extractor with a reflux condenser was set up and heat source adjusted so that the solvent boils gently and leave it to siphon over for 5-6 hours. After that, the sample was dried with the fat residue and filter paper to achieve a constant weight. And the crude fat was calculated as follows:

$$\text{Crude lipid} = \frac{\text{Lipid weight}}{\text{Weight of sample}} \times \frac{100}{1}$$

Where lipid weight = weight of flask after oven drying –weight of empty flask obtained before extraction started.¹⁸

Determination of crude fiber

The crude fiber was determined by weighing 2 g of starch sample into a 250ml conical flask containing 200ml of H₂SO₄. Gentle heat was applied for 30minutes. The flask was maintained to 200ml mark with warm distilled water. It was filtered using a Whatman filter paper and washed to neutrality using distilled water testing with litmus paper. The residue was later washed with 1.25ml of NaOH into the conical flask made up to 200 ml mark, heated gently for 30 minutes with the addition of warm distilled water to maintain 200 ml volume. It was filtered after 30 minutes and rinsed severally with hot distilled water and later with 10% HCL. The residue was put into crucible and dried to constant weight in muffle furnace for 300 °C for 30mins, removed and cool in a desiccator and allowed subsequently to cool at room temperature and weighed.¹⁸

$$\text{Crude fiber} = \frac{\text{Furnance weight} - \text{oven weight (ash)}}{\text{Weight of sample used}} \times \frac{100}{1}$$

Determination of Nitrogen free extract

The Nitrogen Free Extract (N.F.E) was done by subtraction method.¹⁸

N.F.E = 100 – crude protein+ Ash + Lipid + Moisture content.

Determination of phosphorus

Phosphorus was determined by acid digestion using ammonium molybdate. The concentration of phosphate has a direct effect on the yellow hue that appears.¹⁸

Determination of Granular shape and size

Granular shape and size distribution of different starches was performed by using an image analyzer system 'AXL LABO' (Digital tri-nocular microscope), Germany, Labo. A starch sample was sprinkled on the glass slide. When the starch was mixed with the 1:1 water-glycerin solution, 1-2 drops were added, stirred and glass cover slip was then applied to the slide. The slides were observed in optical microscope and images under normal light were obtained. The images selected were analyzed. Magnifications used were x100 and x400. The parameters evaluated were shaped large and small diameter, three slides for each samples.¹⁸

Determination of the iodine

Iodine spectra of the native starches were determined by dispersing starch in 90% DMSO solution in boiling in a water bath.⁸ 20 minutes in a boiling water bath and cooling to room temperature were used to prepare a sample of starch. Allotments of 0.5% of the dispersion were transferred in triplicates to a 25 ml volumetric flask, mixed with about 20 ml of distilled water and 1 ml of iodine-potassium iodide in distilled water solution and filled to the mark. The mixture was stood for 20 min at room temperature and the iodine affinity was determined using a spectrophotometer.

Determination of Amylose content

Amylose content analysis by dividing the starch's iodine affinity by 20%, the amylose content was determined.¹⁸

Synersis characteristics

Synersis characteristics was expressed as the volume of starch separated from water in the starch gel under storage at refrigerator (4°C) temperature for five days. Synersis of gels from starch pastes was determined.²² Starch paste was prepared by heating starch suspension (2.0 g starch in 40 ml of distilled water) at 95°C for 10 min with constant stirring. Gels were cooled stored at refrigerator (4°C) and the weight of the starches recorded for five days.

Statistical analysis

Statistical analysis was computed using SPSS (Statistical Package for Social Science) and Microsoft Excel Statistical Tool-Pack.

Results and Discussion

The amylose content affects gelatinization and retrogradation properties, swelling power and enzymatic susceptibility of starches. Low amylose content was observed in cassava, sweet potato and cocoyam at 20.5±2.50%, 21.8±4.37%, 20.0±2.05% respectively and higher values of 29.8±3.41%, 30.9±2.26% and 24.6±1.04% were obtained in white yam, yellow yam, water yam respectively (Table 3) are in agreement with previous observations in Ivory Coast.²³ Under the light microscope, results of morphological appearance and size of cocoyam, white yam, yellow yam, water yam, sweet potato and cassava starches as determined by AXL LABO are presented in Plates 1 to 6. Cocoyam starch had small and medium-sized granules that were polyhedral (polygonal) and had smooth surfaces, with some areas of the surface being rougher than others (Plate 2). Though, the shape of the granules were quite variable among the tuber starches, cassava, sweet potato, white yam, yellow yam, water yam showed similar granules (from 13.16 to 48.89 µm). The granules of these starches were round and polygonal in shape as shown in (Plates 1, 2, 4, 5 and 6). Granular shape and size are very important characteristics for the starch extraction industry.

Previous workers recorded similar values which ranged from 13.38 to 57.01 µm.²⁴⁻²⁵ Because of the substantial amount of water lost during

retrogradation, starches including potato, cocoyam, and cassava, which have a low amylose concentration, exhibited strong syneresis. There was a continuous increase in syneresis values of gels in the different tuber starches studied (Figure 1).

The low value obtained may be attributed to high amylose content in white yam, yellow yam and water yam. Gel retrogradation is affected, in part, by how the starch chains in the gel are arranged and how they interact with one another during storage. For the five days of refrigerated storage applied, opacity of the cocoyam starch gel significantly increased compared with other starches indicating higher retrogradation tendency in the course of storing (Figure 5). These results line up with what has been seen before.²⁶ Phosphorus is an important non carbohydrate constituent of starch present in tuber starches mainly as phosphates monoester, high phosphorus content in these starches contribute to increase viscosity and lightness. From (Table 3), cassava and water yam have lower phosphorus content (0.008±0.04% and 0.009±0.04%) respectively, while sweet potato and white yam displayed intermediate values of 0.017±.02 % and 0.020±0.05 % respectively. The phosphorous values in cocoyam and yellow yam starches showed higher values (0.024±0.02 % and 0.025±0.05 %) respectively. These values are slightly higher than previous values²⁷ but in agreement with the observations of other studies.^{28, 17} The result for crude fiber was the same for all the starches analyzed (Table 3), as no crude fiber was found which may be due to the extraction process employed. From (Table 2), cocoyam had the highest viscosity which may indicate a greater structural rigidity in comparison to other starches. Cocoyam (0.8) while yellow yam, white yam, cassava were of the order (0.60, 0.45, and 0.35) while water yam and sweet potato had lower values (0.25 and 0.20). These findings are equally in line with previous observations.²⁹⁻³⁰ The higher the temperature, the more the swelling power of the starch which enhances the rigidity of the gel (Figure 1). This also agrees with the observation of other works.^{15, 29}

Table 1: Characteristics of starch solution

Sample	Texture	Transparency	Sterring proof	Retro gradation
Potato	Smooth	Clear	Low medium	Medium
Yellow yam	Smooth	Opaque	Low medium	Medium
White yam	Smooth	Clear	Low medium	Medium
Water yam	Smooth	Clear	Low medium	Low
Cassava	Particles	Clear	Low – high	High
Coco yam	Smooth	Opaque	Low – high	High

Table 2: Physiochemical analysis of the crude starch

Sample	Viscosity	Density	PH	WBC
Sweet	0.200 ± 0.01	0.997 ± 0.05	6.15 ± 1.58	0.92 ± 0.17
Potato				
Yellow	0.600 ± 0.02	0.993 ± 0.05	6.90 ± 1.03	1.15 ± 0.09
yam				
White	0.450 ± 0.04	0.993 ± 0.05	6.50 ± 1.45	0.90 ± 0.25
yam				
Water	0.250 ± 0.04	0.996 ± 0.04	7.00 ± 1.0	0.93 ± 0.30
yam				
Cassava	0.350 ± 0.02	0.998 ± 0.03	6.75 ± 0.78	0.99 ± 0.26
Coco	0.800 ± 0.04	0.999 ± 0.02	6.58 ± 0.92	1.12 ± 0.06
yam				

N value = 3 WBC= water binding capacity

Table 3: Proximate analysis of the crude starch

Sample	Ash%	Moisture %	Crude Protein %	Lipid%	Phosphorus %	N.F.E %	Amylose %	Crude fiber %
Sweet potato	0.18 ± 0.01	9.20 ± 0.03	0.16 ± 0.01	0.12 ± 0.02	0.017 ± 0.02	90.34 ± 0.04	21.8 ± 4.37	0.00
Yellow yam	0.23 ± 0.02	5.00 ± 0.5	0.20 ± 0.02	0.19 ± 0.02	0.025 ± 0.05	94.38 ± 2.03	30.9 ± 2.26	0.00
White yam	0.15 ± 0.01	6.50 ± 1.0	0.10 ± 0.02	0.12 ± 0.02	0.020 ± 0.05	93.13 ± 1.04	29.8 ± 3.41	0.00
Water yam	0.13 ± 0.02	10.0 ± 2.0	0.07 ± 0.02	0.22 ± 0.03	0.009 ± 0.04	89.58 ± 4.02	24.6 ± 1.04	0.00
Cassava	0.19 ± 0.01	8.00 ± 0.5	0.21 ± 0.05	0.17 ± 0.02	0.008 ± 0.04	91.43 ± 1.65	20.5 ± 2.50	0.00
Coco yam	0.21	7.10 ± 1.90	0.18 ± 0.02	0.16	0.024 ± 0.02	92.35 ± 2.22	20.0 ± 2.05	0.00

N value=3 NFE= nitrogen free extract (i.e. carbohydrate content)

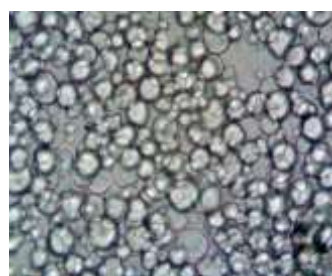


Plate 1a: Cassava ×400



Plate 1b Cassava x 100



Plate 2a: Cocoyam ×400



Plate 2b: Cocoyam ×100



Plate 3a: Sweet potato ×400



Plate 3b: Sweet potato ×100



Plate 4a: Water yam ×400



Plate 4b: Water yam ×100

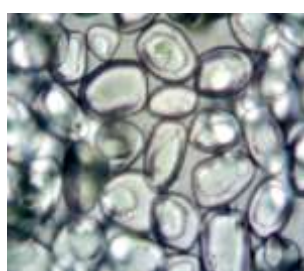


Plate 5a: White yam ×400

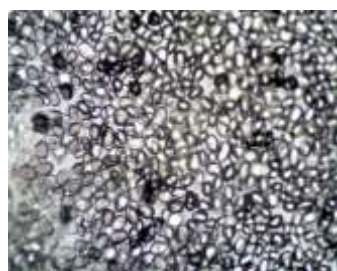


Plate 5b: White yam ×100



Plate 6a: Yellow yam ×400



Plate 6b: Yellow yam ×100

Granular/microscopic shapes of the different tubers

The swelling power of starch is often related to their protein contents. A higher protein content in starch may cause starch granules to become embedded, limiting starch access to water and limiting swelling power. The water binding capacity (WBC) (Table 2) was generally the same for all the starches analyzed though, on average, high for yellow yam and cocoyam (1.15 and 1.12 respectively) and low for cassava, 0.99, sweet potato, 0.92, water yam, 0.93 and white yam, 0.93 starches. The WBC is highly dependent on the crystalline properties of starch being high for starches with low crystallinity and hence depends on the associated forces among starch components where weak inter associative forces result into high WBC³¹⁻³². In food industries, the amount of water taken up by starch is very important. This depends on the type of starch and its botanical sources. In this study, no significant differences ($p > 0.05$) were observed for WBC, although, differences could occur in individual tuber varieties. This gives premise for variety

specific selections for this parameter in addition to modification of starch, particular tubers to meet the application demands. Among the starch samples, cassava had the highest percentage of protein content (0.21 ± 0.05), yellow yam (0.20 ± 0.02), cocoyam (0.18 ± 0.02), sweet potato (0.16 ± 0.01) while white yam and water yam had the lowest amount of 0.10 ± 0.02 and 0.07 ± 0.02 respectively. Previous researchers observed similar findings.³³⁻³⁴ Protein content varies because starch types vary in structure. However, the results obtained in this study were lower than those reported previously for yam, sweet potato 55, 36%; 0.14%³⁵⁻³⁶ and cocoyam (20.9-40%).³⁷ While the difference might have been caused by a different extraction method, it could also be due to the variations of botanical origin and environmental conditions during cultivation. The mineral, physical and chemical composition of soils affect the performance quality of soils used for agricultural purposes.

These factors determine the ion exchange and water retention capability of the soil. Availability of essential micronutrients in the right proportion in the soil are important for proper growth of plants. The predominant substances in the soils in Benin City are kaolinite, feldspar, quartz and sesquioxides of aluminium, iron and goethite. The soil is acidic with very low cation retention and buffering capacities.³⁸ It is for these reasons that the proximate contents of edible tubers grown in this area were studied.

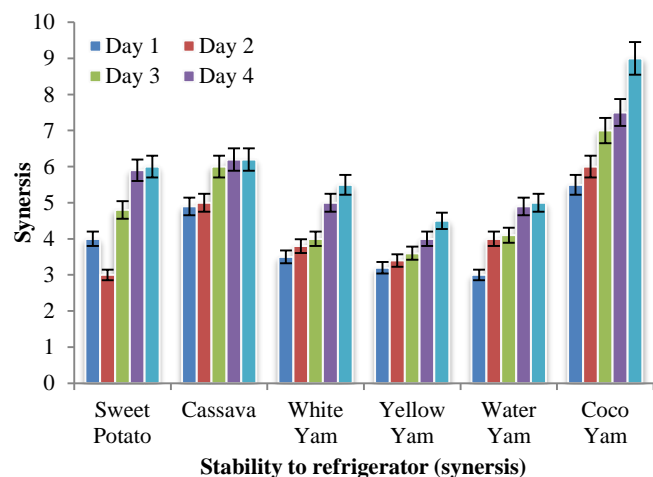


Figure 1: Stability of the starch of edible tubers to refrigeration (syneresis)

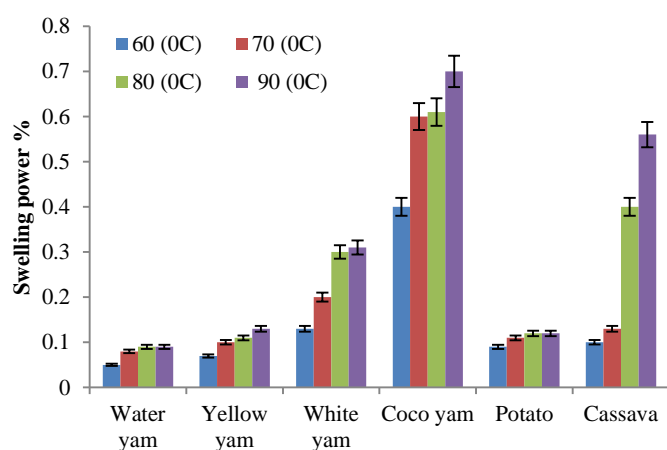


Figure 2: Swelling power of the starch in edible tubers during refrigeration

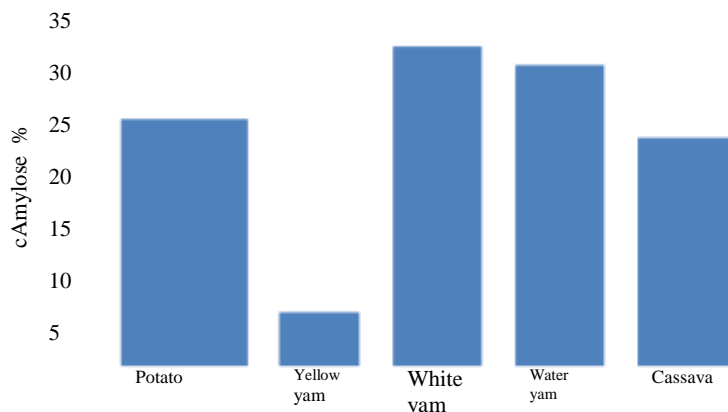


Figure 3: Amylose content in the edible tubers

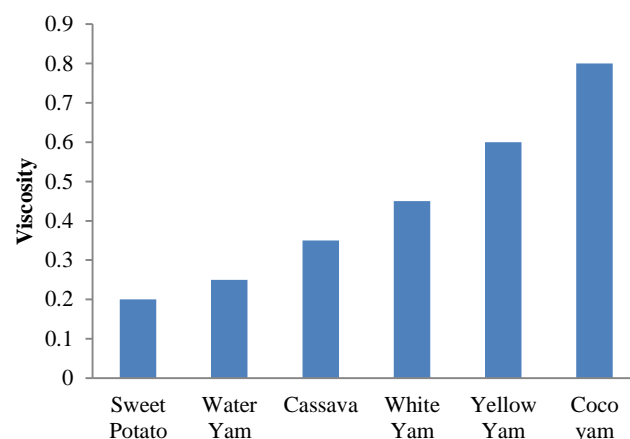


Figure 4: Viscosity of starch in the edible tubers

Conclusion

From our findings, the physicochemical characteristics of the starches extracted from the six edible tubers would enhance their potentials in food processing industries. Also, these qualities will improve their nutritional values to humans. This will allow for informed choices or diversity of choice when sourcing new ingredients with properties to enhance product production and development. The high viscosity of cocoyam and yellow yam starches makes them very useful in food applications where high thickening power is desired. The stability against heat and chemical treatment would also be useful in many food applications. The low viscosity of sweet potato, white yam and water yam flour is desirable in the food industry for applications that require lower viscosity and the high paste clarity would make it useful for products where this is required as a thickening agent. Therefore these tubers may be seen as having very broad applications within the food industry.

Conflict of Interest

The authors declare no conflict of interest.

Authors' Declaration

The authors hereby declare that the work presented in this article is original and that any liability for claims relating to the content of this article will be borne by them.

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