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Morphological Characterisation and Analysis of Functional Properties of Yellow and White Biter Yam (*Dioscorea dumentorum*) Derived Starches

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Abstract: The study focused on the extraction of starches from white and yellow bitter yam (*Dioscorea dumentorum*) tubers with a view to comparing their properties to other conventional starches. Morphological characterisation and functional properties of the two starches extracted from two varieties of bitter yams have been determined using standard analytical procedures. The starch samples were subjected to scanning electron microscopy (SEM) analysis coupled with Energy dispersive x-ray spectroscopy (EDS) analysis and Fourier Transform Infra-Red (FTIR) spectroscopy analysis. The starch yields fell within the appreciable quantity range which is important for commercial purposes, and the moisture content ranked within the stipulated range which is good enough for storage ability. The swelling capacity, bulk density, and ash content compared relatively well with other conventional starches in the literature. The surface morphology of white bitter yam starch was observed to be smoother than that of yellow bitter yam starch. The EDX analysis revealed that the elemental compositions of the two starches were dominated by C and O, this showed that the two elements are the major components in the chemical framework of the obtained starch. The FTIR analysis revealed the functional groups expected from starch. It can be opined that white and yellow bitter yams can be alternative starch sources for the industrial world as this will ease the pressure on the convectional starch sources.

Keywords: Bitter Yam, Starch, Morphological Characterization, Swelling Capacity, Spectroscopy

1. Introduction

Tubers and root crops are very important and are ranked closely with cereals as significant sources of carbohydrates in the world [1]. Different tuber crops are often taken with nutritional and non-nutritional values [2]. Tubers and root crops are rich sources of carbohydrates, and this has invariably improved their consumption as staple foods. Also, their non-application as food cannot be overemphasised simply because of the considerable starch content. Hence, an impetus for using their starches in starch-based industries compared to some other polysaccharides [3].

Yam *(Dioscorea spp*) is a very important tuber as it serves as food with so much socio-economic importance in many tropical countries [4]. However, bitter yam (*Dioscorea dumetorum),* an underutilized yam variety, belongs to the family *Dioscoreaceae* and genus *Dioscorea* [5]. As an underutilised yam variety in many African countries, including Nigeria, Bitter yam can be propagated and cultivated, and it may also grow freely in the bush [6]. The underutilisation and perhaps low consumption of bitter yam may be partly attributed to the bitterness in taste of the tubers and hardening of the texture, which occurs when in storage [7]. Despite all the limitations to its broader uses, it still possesses some characteristics that can be harnessed and utilised for better applications.

Starch is undoubtedly the most important carbon reserve in all plants as contingent upon the quantity, various distribution within different varieties of plants, and its economic importance [8]. It comprises glucose polymers of varying

degrees ordered in a three-dimensional and semi-crystalline matrix or granules. Starch is among the relatively abundant carbohydrate polymers that are natural, so it is generally considered safe and low cost. These properties give it wider publicity for industrial applications [9].

There have been some reports on isolation, analysis, and utilization of starches obtained from yam [10] and bitter yam (although scanty) [11-14].

As crucial as starches are in food and non-food industries, popular starch sources have always been potatoes, corns, etc. However, the growing demands for starch on a global scale need to be considered. To ease the pressure on the existing starch sources, it is essential to look for other sources, such as underutilised bitter yam tubers, the more reason for this study.

2. Materials and Methods

2.1. Sample Collection

White and yellow bitter yam (Dioscorea dumentorum) tubers were obtained from a local market in Ado-Ekiti, Nigeria. All the reagents, such as HNO_3, HCl, etc., were of analytical grade, and they were obtained from the Chemistry Laboratory of Ekiti State University, Nigeria.

2.2. Extraction of Starch

The tubers of each variety were adequately washed with clean water to remove soil particles and other debris. The tubers were peeled, chopped, grated, and properly blended, 100g of the blended sample was mixed with 500 mL of distilled water (ratio 1:5), and the slurry was sieved using a muslin cloth. This product was allowed to settle overnight to give two layers (sediment and supernatant), and the supernatant was decanted to obtain starch in its crude form. The obtained starch was washed severally by dispersing it in distilled water and subsequently allowed to settle. The resulting starch mash was oven-dried at 50°C until a constant weight was obtained. The dried starch was sieved into an air-tight plastic container and stored at room temperature before further analysis.

2.3. Percentage of Starch Yield

The percentage yield was determined by expressing the weight of isolated starch as a percentage of the weight of fresh tubers. This can be expressed as follows:

$$\text{Yield (\%)} = \frac{W1}{W2} \text{ x } 100$$

Where W_2 = weight of fresh tubers and W_1 = weight of dried starch isolated.

2.4. Moisture Content

The moisture content was determined using a method described by Olayemi et al. [15]. A crucible was washed, oven-dried, and cooled in a desiccator, and the weight was denoted as W_1. About 5 g of the starch sample was placed in the crucible, and the combined weight was denoted as W_2. The crucible with the sample was then oven-dried at 105°*C* for 3 h. After the set time, the crucible was removed, cooled in a desiccator, and weighed. The drying, cooling, and weighing processes were repeated until a constant weight (W_3). The percentage moisture was calculated as follows:

$$\%Moisture\ content = \frac{weigt\ loss}{weight\ of\ sample}\ X\ 100$$

$$\%Moisture\ content = \frac{W_2 - W_3}{W_2 - W_1}\ X\ 100$$

2.5. Ash Content

The ash content of each starch sample was determined usingthe method described by Ohwoavworhua *et al*. [16]. Five grams (W_0) of starch was measured into a porcelain crucible which had been thoroughly washed, pre-heated, cooled, and weighed (W_1). The crucible with its content was heated to 550°C in a muffle furnace and left for 3 h to ash. After that, the crucible was removed from the furnace, cooled in a desiccator, and weighed (W_2). The total ash content was calculated and expressed in percentage. This analysis was carried out in replicates.

$$\%Ash\ content = \frac{W_1 - W_2}{W_0}\ X\ 100$$

Where; W_2 is the weight of crucible + sample after ashing; W_1 is the weight of crucible + sample; W_o is the weight of the sample.

2.6. Bulk Density and Tap Density

Bulk and tap densities of each of the samples were determined by weighing 5 g (*W*p) of the starch powder and carefully transferring it into a dry 100 mL graduated measuring cylinder. The volume occupied by the starch sample without tapping was denoted as *V*p. The cylinder was then continuously tapped on a wooden surface at a certain height until there was no further reduction in the volume; this volume was taken as the tapped volume (Pvt). The densities were determined in replicates [15].

The bulk (B_d) and the tapped densities (T_d) were calculated as follows:

$$\text{Bulk Density (\%)} = \frac{W_p}{V_p} X\ 100$$

$$\text{Tap Density (\%)} = \frac{W_p}{V_pT} X\ 100$$

Where *W*p is the weight of the sample used, *V*p is the volumeof the sample without tapping, *V*p*T* is the volume of the sample after tapping.

2.7. Swelling Capacity

Accurately measured 1 g of each starch sample was measured and denoted as *V*x. The measured quantity was dispersed in distilled water (10 mL). The mixture was left standing for 24 h, and the volume of the sediment was taken and denoted as *V*v. The swelling capacity was computed as follows:

$$\%Swelling\ capacity = \frac{Vv - Vx}{Vx}\ X\ 100$$

Where *V*v is the volume of sediment after 24 h; *V*x is the volume of the sample.

2.8. Characterisation of Starch Samples

Each starch sample was analysed using Scanning Electron Microscopy (SEM, JSM 5910LV, Japan) coupled with Energy Dispersive Spectroscopy (EDS) and Fourier Transform Infra-red Spectrometer (Thermo Scientific, Nicolet IS50 FTIR, USA).

3. Results and Discussion

The percentage starch yields of white and yellow bitter yams were 26.35 and 25.13%. The result showed that the percentage starch yield of white bitter yam was more than its yellow variety. Therefore, the percentage yields obtained in this report aligned with the report of Akinoso and Abiodun [12], which illustrated that the percentage yield of white bitter yam starch was higher than that of yellow bitter yam. The percentage yield of starch obtained from bitter yam could range from 11 to 88%. This range is contingent upon the different methods of extraction and the varieties of tubers [12, 13].

3.1. Functional Properties of the Two Starches

The results of the functional properties of the two starches are presented in Table 1.

Table 1. *Functional properties of white and yellow bitter yam starches.*

	White bitter yam starch (%)	Yellow bitter yam starch (%)
Moisture content (%)	0.863± 0.012	0.92 ± 0.057
Ash content (%)	0.93± 0.012	0.82±0.02
Bulk density (%)	0.44±0.058	0.45±0.057
Tap density (%)	0.50±0.02	0.55±0.21
Swelling capacity (%)	2.67± 0.23	1.78±0.11

The moisture contents for the white and yellow bitter yam starches were 0.863±0.012 and 0.920±0.057%. The results showed that the moisture contents for the two starches were generally low. However, it is worthy of note that the moisture content of white bitter yam starch was lower than the value obtained for yellow bitter yam starch. Makanjuola and Makanjuola [17] reported that the moisture content of bitter yam starch was lower than the moisture contents obtained from water yam and white yam starches. The low moisture content in this work was in line with the reports of Ashworth and Draper [18]. Low moisture content in starch is a good signal for storage ability. A high level of moisture could allow microorganisms to breed, which will invariably lead to the deterioration of the materials [19].

The ash contents were 0.930±0.012 and 0.820±0.02% for white and yellow bitter yam starches. It has been reported that the ash content (0.02 - 0.09%) of bitter yam starch may be present in low quantities, and the low ash content of the starch has been taken as a basis for starch purity [20, 21]. Low ash contents in starch are due to the unavailability of fine fibres (hydrated) linked to the cell wall of starch [22].

The bulk density value for white bitter yam starch was 0.440±0.058%, while the bulk density for yellow bitter yam starch was 0.450±0.057%. The bulk density values for the two starches (white and yellow bitter yam starches) were relatively close. Bulk density is dependent on the size of particles and density, and it plays a good role in packaging and handling requirements [23]. Bulk density can be directly linked to the heaviness and density of a starch sample which denotes that the volume of material will not drastically reduce under storage [17]. The tap density values for white and yellow bitter yam starches were found to be 0.500±0.02 and 0.550±0.210%, respectively.

The swelling capacity value for white bitter yam starch was 2.670±0.23%, while it was 1.780±0.11% for yellow bitter yam starch. The swelling capacity values for the two starches under investigation fell within the range reported by [12] for white and yellow varieties. Swelling capacity is the volume of expansion of molecules in response to water uptake, which it possesses until a colloidal suspension is achieved or until intermolecular forces prevent further expansion and uptake in the swollen particles [24]. Differences in the swelling capacity of starches obtained from bitter yams could be due to varying experimental procedures, the ratio of starch to water, the nature of the cultivar, and perhaps the induced temperature [25].

3.2. Characterisation of White and Yellow Bitter Yam Starches

3.2.1. SEM Analysis of the Samples

The samples (white and yellow bitter yam starches) were subjected to SEM analysis, and the results are presented in Figure 1. The SEM images of white and yellow bitter yam starches, as presented in Figure 1 showed that the white bitter yam starch possessed some fine and smoother surfaces having distinct granulated shapes of different sizes. In contrast, yellow bitter yam starch morphology had rough and pebble-like structures. The SEM micrograph of the white bitter yam starch under study resembled the micrograph reported by Odeku and Picker-Freye [26]. Shujun *et al.* [27] reported that the SEM micrograph of white bitter yam starch appeared in small rods that were round and agglomerated in shape, while the micrograph of yellow bitter yam starch appeared in the form of rocks of different shapes having rough surfaces. The absence of cracks and other thin lines on the micrographs of starch morphology is also used as a yardstick for starch purity [28].

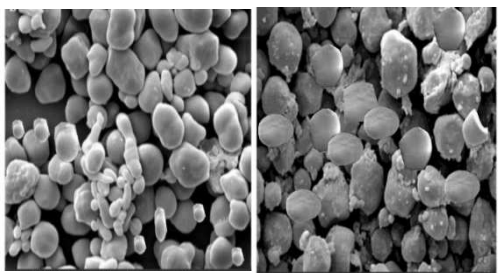

Figure 1. *SEM micrographs of white and yellow bitter yam starches.*

3.2.2. EDS Analysis of the Samples

The EDS images for the white and yellow bitter yam starches are shown in Figure 2. The identified minerals in their percentages for white bitter yam starch were O (35.00%); C (60.20%); S (4.10%); Si (0.30%); and Na (1.20%). For yellow bitter yam starch, the mineral components observed in the micrograph were O (26.45%); C (63.07%); Si (6.78%); S (2.20%); and Ca (1.50%). The results showed that C and O were the major components in the starch structure. Silicon and Sulphur were found in the two starches under investigation. However, sodium (Na), although in a trace amount, was detected in white bitter yam starch, unlike yellow bitter yam starch, whereas calcium was present in yellow bitter yam starch and was below detection level in white bitter yam starch. The presence and absence of either Na or Ca in the two starches could be due to different mineral components present in the soils where the tubers were cultivated [29], the method of planting, climatic conditions, and harvesting procedures.

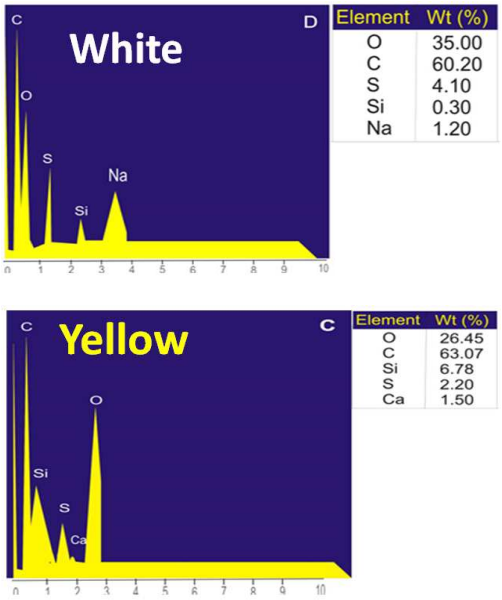

Figure 2. *EDS images of white and yellow bitter yam starches.*

3.2.3. FTIR Analysis of the Samples

The FTIR results for white and yellow bitter yam starches are presented in Table 2. FTIR analysis is often used to identify the major functional groups present in a particular starch sample.

Table 2. *Notable bands of the FTIR analysis for white and yellow bitter yam starches.*

WBYS (cm^{-1})	YBYS (cm^{-1})
3428.00	3426.00
2901.57	2256.00
1635.47	1644.00
1439.90	1199.82
1157.33	1050.00
1102.36	880.20
1048.42	581.00
901.00	416.25
600.36	381.49
448.00	-

*WBYS- white bitter yam starch; YBYS- yellow bitter yam starch.

For white bitter yam starch, a band at wavelength 3428.4cm^{-1} was observed. This band could be assigned to N-H stretch, the hydroxyl group stretch (O-H bond), and bound water peculiar to starches [30]. A prominent peak was observed at 2901.57 $cm^{-1,}$ which indicated C-H stretch. Vibrations at 1102.36 cm^{-1}, 1048.42 cm^{-1}, and 901 cm^{-1} bands were also observed for white bitter yam starch. Smits *et al.* [31] reported that the wavelength at 1047 cm^{-1} connoted ordered regions while the band at 1022 cm^{-1} indicated disordered regions of the starch. The results in this study aligned with the submission of Adewumi *et al.* [32], who reported that the peaks at 3394.72 cm^{-1} and 2929.87 cm^{-1} denoted O-H and C-H stretches, respectively, while the bands in the range of 1654.92 - 1637.56 cm-^{1} and 1438.18 cm^{-1} represented O-H and C-H bending. A band was noticed for yellow bitter yam starch at vibration 3426 cm^{-1} representing O-H stretch. This band resulted from the bound water, which is common to all starches. Bands at wavelengths 1644.92 cm^{-1} and 1199.82 cm^{-1} were assigned to C=C and C–O stretches, respectively. These reports corresponded to O-H and C-O-C functional polysaccharide groups, as reported by Mano *et al*. [33].

The difference in the wavelengths of the absorption bands between white and yellow bitter yam starches might be due to environmental factors such as exposure degree to sunlight and components within the soil where the tubers were cultivated.

4. Conclusion

The observed properties of the two starches which are relatively adequate as obtained in this study are good signals that they can be harnessed and utilized in many starch related industrial set-up. The results compared reasonably well with the functional groups and instrumental analyses of some starches from other conventional sources as documented in literature. Hence, it can be said that white and yellow bitter yams (*Dioscorea dumetorum*) tubers are good sources of starch for many industrial applications.

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