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Original Article

Physicochemical and material properties of starches from three cultivars of *Dioscorea rotundata*

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ABSTRACT

Dioscorea rotundata (white yam, guinea yam or African yam) is the most important species of yam in West Africa because of its economic value and uses. Starches from three cultivars of *Dioscorea rotundata* namely, *Giwa, Lagos, and Sule,* were characterized for their physicochemical and material properties. The starches were evaluated using elemental and proximate analyses, scanning electron microscopy, X-ray powder diffraction analysis and density measurements. The results showed that the starch granules from the three cultivars of *D. rotundata* were oval-oblong in shape with a size range of 3.19–3.59 μm. The X-ray diffraction pattern of the starches from the three cultivars was of the C– type with relative crystallinity of 21%. The results showed that starches from the three *D. rotundata* cultivars had similar proximate, physicochemical and material properties. The starches had similar functionality, probably due to the similarities in their botanical sources. Thus, starches from the three cultivars of *D. rotundata* could be interchangeable as excipients in pharmaceutical tablets.

Introduction

Yams are staple root crops cultivated in West and Central Africa belonging to the genus *Dioscorea,* which includes over 600 species, with the most cultivated and economically important species being *Dioscorea rotundata* Poir, *D. dumetorum*; *D. oppositifolia* Thunb; *D. alata* DIAL2 and *D. bulbifera*, *D. esculenta* Lour and *D. cayenensis* Lamk (Coursey, 1967). The edible species are rich in starch (70–80% dry weight basis) and are usually consumed in different forms resulting from any of the processes of boiling, drying, fermentation, frying, milling, pounding, roasting and steaming (Odeku, 2013). Yams are widely grown in sub-Saharan Africa with Nigeria being the largest producer (Food and Agricultural Organization, 2013). Nigeria accounted for over 65% (38 million t) of the world yam production, valued at $7.75 billion and cultivated about 2.9 million hectares of land in 2012 (www.iita.org/yam). However, yam tuber loss in storage in Nigeria has been reported in the range 30–66% of the total output (Ugwu, 2009). Such losses have been associated with pests, diseases, damage during and after harvest (during transportation) and temperature and humidity variations, as well as to varietal differences (Asadu et al., 2008).

Among the *Dioscorea* species, *D. rotundata* (white yam or guinea yam or African yam) is the most important in West Africa because of its economic value and uses (Degras, 1993). Studies have shown that starches obtained from the edible species of yams could find application in the food and pharmaceutical industries due to the yams' physicochemical and material properties (Okunlola and Odeku, 2011a; Odeku, 2013). Starches from the different *Dioscorea* species were found to be suitable as excipients that could be useful for commercial production of pharmaceutical tablets (Odeku and Picker-Freyer, 2007; Okunlola and Odeku, 2011b. However, more than 20 cultivars of *D. rotundata* have been identified in Nigeria (Odu et al., 2004). With the proof of the usefulness of the starches obtained from this species, it is of interest to investigate the starches from the different cultivars of white yam, particularly to correlate the properties of the different varieties found within Nigeria with different local names with the view to classifying them based on their similar properties and use as excipients. This is particularly important since the properties and functionality of starches have been shown to vary, probably due to differences in their botanical sources as well as the biosynthesis of the starch granules and the physiology of the plants (Odeku, 2013).

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In the present study, the proximate composition, physicochemical and material properties of starches obtained from three local varieties of *Disocorea rotundata* (*Giwa, Lagos and Sule*) were evaluated to determine their relative suitability and interchangeability as excipients in the food and the pharmaceutical industries.

Materials and methods

Tubers of three cultivars of *Dioscorea rotundata* Poir, widely found in Nigeria, namely *Giwa, Lagos,* and *Sule*, were harvested fresh from the farm in Pmazi village in Bosso, Minna, Niger state, Nigeria, and authenticated. The starches were extracted from the relevant tubers using established procedures (Okunlola and Odeku, 2011b. Briefly, fresh tubers of yam were washed, peeled and then cut into small pieces. The pieces were then washed with sodium metabisulphite in distilled water to prevent darkening and then milled into a fine paste using a laboratory mill. The slurry was strained through a muslin cloth and the filtrate was left to settle. The supernatant was decanted at 12 h intervals and the starch slurry re-suspended in distilled water. The starch cake was collected after 3 d and dried in a hot-air oven at 60 °C for 48 h. The dried mass was pulverized using a laboratory blender and then screened through a 120 μm mesh sieve (Young, 1984; Okunlola and Odeku, 2010b).

Elemental analysis

The elemental analysis and microstructure of the starches were carried out using an X-ray fluorescence (WDXRF) spectrophotometer for elemental analysis (Rigaku Supermini WD-XRF; Rigaku Cooperation; Tokyo, Japan) at 50 kV/200 W.

Proximate composition

The crude fat, crude fibre, ash and lipids contents were determined according to the methods of Association of Official Analysis Chemists (2000). The crude protein content was estimated from the nitrogen content determined by elemental analysis using a conversion factor of 6.25 (Gebre-Mariam and Schmidt, 1998). The free sugar content was quantitatively determined for each starch material using the method described by Dubois et al. (1956). The amylose content was determined calorimetrically using the method described by Williams et al. (1970). All determinations were done in triplicate and the results shown as mean ± SD. Significant differences were tested at $p < 0.05$.

X-ray powder diffraction

The X-ray diffraction was recorded with Cu-$K_{\alpha 1}$ at 25 °C using a Philips X'pert pro (PANalytical B.V.; Eindhoven, the Netherlands) at a voltage of 40 kV 30 mA. The starch powder was tightly packed in a sample holder and the scanning region of the diffraction angle (2θ) was 2–50°.

Material properties

Scanning electron microscopy

The starch powders were sputtered with gold and analyzed in a scanning electron microscope (ESEM 30; Philips; Kassel, Germany) at an accelerating voltage of 2 kV.

Particle size distribution

The particle size distribution of the starches was determined using optical microscopy (Laborlux II; Leitz; Wetzlar, Germany) with approximately 300 particles for each starch.

Density measurements

The apparent particle densities of starches were determined using a helium pycnometer (Acupye 1330; micromeritics; Norcross, GA, USA).

Bulk and tap densities were determined in a 250 mL cylinder using a volumeter (Stamp volumeter Stav, 2003; J. Engelsmann AG; Ludwigshafen, Germany). Determinations were also made in triplicate according to European Pharmacopoeia (2007).

Powder flowability

The flowability of the starches was assessed using the Hausner ratio and the Carr index (Carr, 1965; Odeku and Picker-Freyer, 2007).

The flow rate of the starch powders was determined using a steel funnel on a Pharmatest flow rate apparatus (Sartorius Pharmatest; Apparatebau GmbH; Hainburg, Germany) with an orifice of 15 mm.

Swelling power and water binding capacity

The swelling power at room temperature (27 ± 2 °C) was determined using the method described by Leach et al. (1959), while the water binding capacity was determined using the method of Ring (1985).

Results and discussion

Elemental and proximate composition

The elemental composition of starches have been known to play an important role in the physicochemical properties and functionality of starches (Raeker et al., 1998; Siliveru et al., 2017). The results of the elemental analysis are presented in Table 1 and show the absence of heavy metals but the presence of aluminum, selenium, phosphorus, sulphur potassium, calcium and iron in all the species. This result is consistent with the report for starches from another *Dioscorea* species (Odebunmi et al., 2007). Furthermore, the amount of phosphorus present in the *Dioscorea* starches was similar to that reported by Odeku and Picker-Freyer (2007) for starches from tubers of another cultivar (*Dioscorea rotundata* c.v. *Igangan*) grown in South West Nigeria. A high phosphorus content in tuber starch has been shown to affect the physical properties of the starch such as the viscosity, gelatinization temperature and transparency (Aprianita, 2010; Zhang et al., 2017).

The proximate composition of the starches presented in Table 2 indicates that there were no significant differences in the proximate composition of the starches from the different cultivars. There were no significant differences in the amylose content of the starches and the values were similar to those reported for other cultivars of *Dioscorea* starches (Odeku and

Table 1
Elemental analysis of starches in three cultivars of *Dioscorea*.

Element	Cultivar		
	Lagos (%)	*Giwa* (%)	*Sule* (%)
Mg	–	0.011 ± 0.004	–
Al	0.133 ± 0.003	0.122 ± 0.003	0.103 ± 0.003
Si	0.058 ± 0.003	0.049 ± 0.003	0.044 ± 0.003
P	0.035 ± 0.002	0.022 ± 0.002	0.024 ± 0.002
S	0.011 ± 0.001	0.007 ± 0.002	0.006 ± 0.002
K	0.009 ± 0.009	0.071 ± 0.001	0.066 ± 0.001
Ca	0.004 ± 0.001	0.002 ± 0.000	0.003 ± 0.000
Fe	0.001 ± 0.000	0.001 ± 0.001	0.001 ± 0.000
Zn	–	0.001 ± 0.000	–

Table 2
Proximate composition of starches in three cultivars of *Dioscorea*.

Starch	Moisture content (%)	Crude protein (%)	Crude fat (%)	Total ash (%)	Crude Fiber (%)	Crude carbohydrate (%)	Amylose content (%)
Lagos	9.67 ± 0.43	1.23 ± 0.03	0.39 ± 0.04	1.06 ± 0.02	1.08 ± 0.01	86.60 ± 0.02	23.06 ± 0.12
Giwa	9.58 ± 0.32	1.27 ± 0.05	0.43 ± 0.01	1.03 ± 0.03	1.10 ± 0.03	86.61 ± 0.01	23.16 ± 0.30
Sule	9.98 ± 0.13	1.36 ± 0.02	0.44 ± 0.02	1.09 ± 0.03	1.13 ± 0.02	86.02 ± 0.03	22.86 ± 0.42

Picker-Freyer, 2007). The amylose content of starches has been shown to vary depending on the botanical source of the starch, the climatic conditions and the soil type during growth (Brunnschweiler et al., 2005; Okunlola and Odeku, 2011a). The moisture content for all the starches was less than 10%, which is within the limit required for storage of starches to prevent spoilage. The optimum moisture content in starches is important for good flow and compaction properties and also to prevent the deterioration of the excipients and tablets (Odeku and Picker-Freyer, 2007).

X-ray diffraction

The X-ray diffraction pattern of the starches is shown in Fig. 1 while the percentage crystallinity and crystal type are presented in Table 3. These reflection pattern values were interpreted as an intermediate pattern between A and B and they have been referred to as a C-type diffraction pattern, which is not a true crystalline polymorph but rather a mixture of A and B polymorphs (Brunnschweiler et al., 2005). This is similar to the diffraction patterns reported for a different cultivar of *D. rotundata* and other varieties of *Dioscorea* starches (Farhat et al., 1999; Riley et al., 2006; Odeku and Picker-Freyer, 2007).

The result of the relative crystallinity of the starches showed that the starch exhibited a low degree of crystallinity. There was no significant ($p > 0.05$) difference in the relative crystallinity of the starches from the different cultivars. These values are within the range reported for starches from other botanical sources which were shown to range 15–45% (Napaporn et al., 2001).

Material properties

The scanning electron micrographs (SEM) of the starches are shown in Fig. 2, while the material properties of the starches are presented in Table 4. The micrographs show that the shape of the starches from the different *D. rotundata* samples was oval-oblong. These observed shapes were consistent with those reported for the different cultivar of *D. rotundata* (Odeku and Picker-Freyer, 2007). There was no significant ($p > 0.05$) difference in the size of the starch granules from the different cultivars. The physiology of the plants was also similar (although the soil and climate differed in the areas where they were cultivated), as well as the biosynthesis of the starches, which may have been responsible for the small differences observed in the shape and size of the granules.

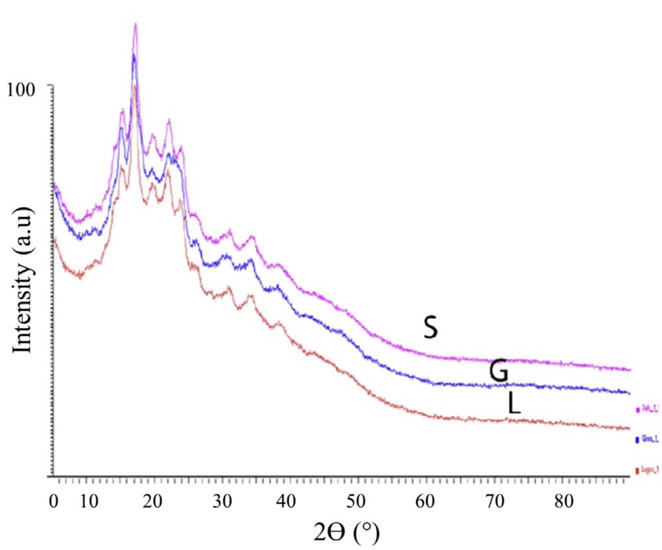

Fig. 1. X-ray powder diffraction patterns of *Lagos* (L), *Giwa* (G) and *Sule* (S) starches.

The results showed that there were no significant differences ($p > 0.05$) in the material properties of the starches from the different *D. rotundata* cultivars. The bulk density of a powder describes its packing behavior during the various unit operations of tableting such as die filling, mixing, granulation and compression (Itiola and Pilpel, 1986; Odeku and Itiola, 1998). A higher bulk density is advantageous in tableting due to a reduction in the fill volume of the die (Odeku and Itiola, 1998). The densities of the starches were very similar and therefore the starches should be handled in a similar manner when used as excipients in tablet formulations. The particle size and shape of have been shown to affect the density of starches (Odeku et al., 2008).

Carr's index has been used as an index of powder flowability and compressibility (Carr, 1965). The lower the Carr index of a material, the better the flowability, but the poorer the compressibility. The Carr's index of the starches was in the range 19–29, which represents fair to poor flow properties (Carr, 1965). Powders with a small particle size have been shown to exhibit poor flowability which may result in problems during die filling and would require a glidant to facilitate powder flow (Odeku and Picker-Freyer, 2007).

The ranking of the swelling capacity was *Giwa* > *Lagos* > *Sule* while the ranking of the water binding capacity was *Sule* > *Giwa* > *Lagos*, although there were no significant ($p > 0.05$) differences in the swelling and water binding capacities of the starches from the different cultivars. This may be attributed to the similarity in the properties such as the amylose/amylopectin content, molecular weight, conformation, degree of polymerization and degree of branching of amylopectin (Ige and Akintunde, 1981).

The results showed that starches from the three cultivars of *Dioscorea rotundata* (*Giwa*, *Sule* and *Lagos*) exhibited similar physicochemical and material properties. Thus, the similar functionality of starches could probably be due to the similarity in their botanical sources and the biosynthesis of the starch

Table 3
Percent crystallinity and crystal type of starches in three cultivars of *Dioscorea rotundata*.

Cultivar	Crystal type	Crystallinity (%)
Sule	C	21.00 ± 0.01
Giwa	C	21.25 ± 0.01
Lagos	C	21.25 ± 0.02

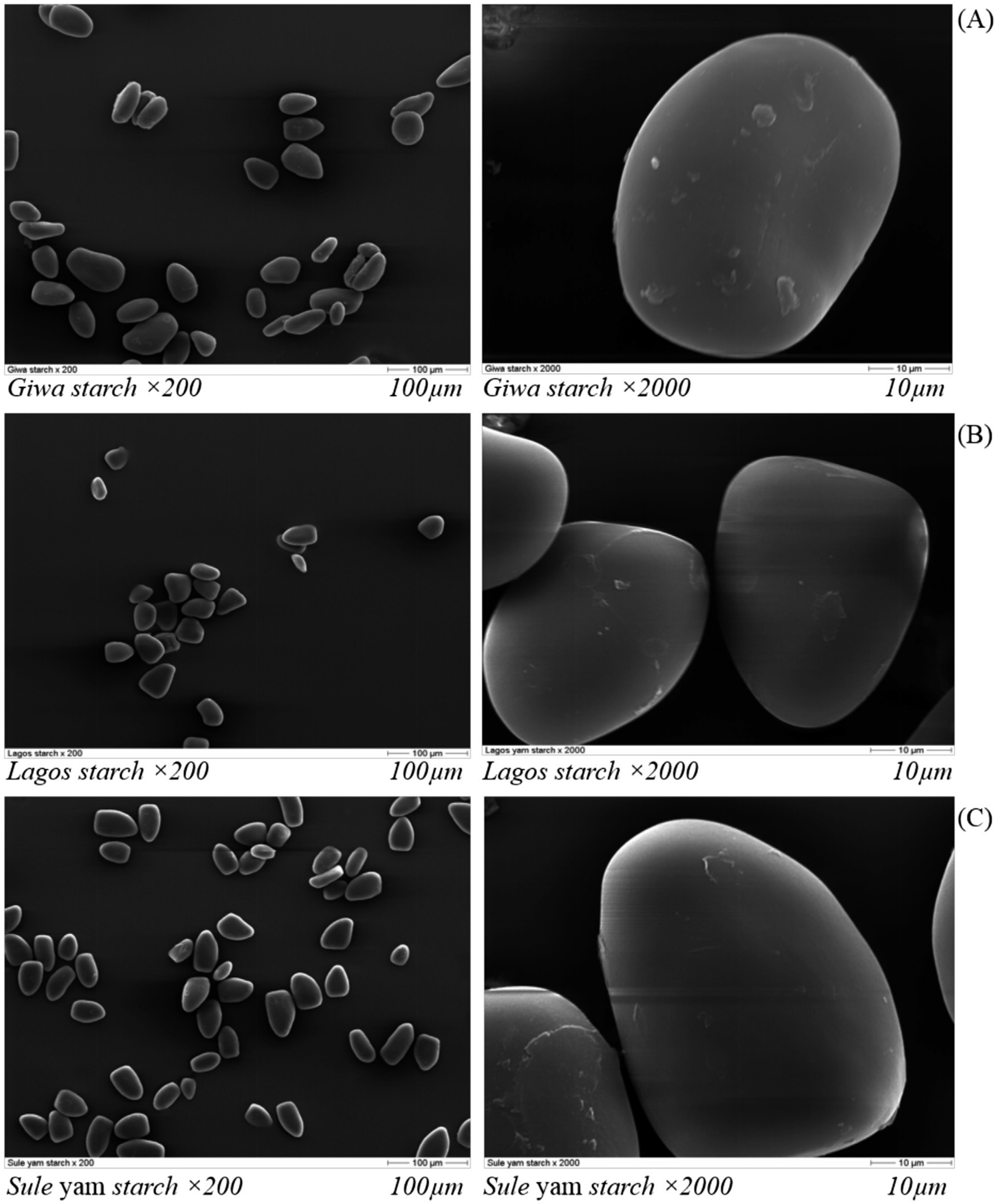

Fig. 2. Scanning electron microscope images (200x and 2,000x) of different cultivars of Dioscorea starches. (A) Giwa starch 200x, 100 μm and Giwa starch 2,000x, 10 μm; (B) Lagos starch 200x, 100 μm and Lagos starch 2000x, 10 μm; (C) Sule yam starch 200x, 100 μm and Sule yam starch 2000x, 10 μm.

Table 4
Material properties of starches in three cultivars of *Dioscorea*.

Cultivar	Particle size (μm)	Particle shape	Apparent particle density (g/cm^3)	Bulk density (g/cm^3)	Tapped density (g/cm^3)	Carr Index (%)	Angle of repose (°)	Swelling Capacity (w/w)	Water binding capacity (w/w)
Lagos	3.54	Oval- oblong	1.59	0.509	0.635	19.84	65.60	1.20	0.57
Giwa	3.59	Oval- oblong	1.57	0.599	0.790	24.17	57.00	1.29	0.67
Sule	3.19	Oval - oblong	1.49	0.506	0.715	28.81	65.90	1.19	

granules. Thus, starches from the different cultivars of *Disocorea* starches could find application in pharmaceutical tablets.

Conflict of interest

The authors declare no conflict of interest.

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