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Physicochemical, functional and antinutritional properties of starches from *Caladium bicolor* and *Dioscorea dumentorum*

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ABSTRACT

Starch samples from the wild species of cocoyam (*Caladium bicolor* (Aiton) Vent.) and three leaf yam (*Dioscorea dumentorum* (Kunth) Pax.) found abundantly in the south and eastern regions of Nigeria, were characterized for their physicochemical, functional and antinutrient properties. *C. bicolor* had higher amylose (17.68%), carbohydrate (83.57%) and ash (2.85%) than *D. dumentorum*. Moisture, crude lipid, protein and fibre ranged between 7.56 to 10.29%, 0.12 to 0.15%, 0.11 to 0.13%, and 2.85 to 3.39%, respectively. The starches exhibited promising functional properties with *D. dumentorum* having higher dispersibility (64.85%), solubility (73.99%) and oil absorption capacity (2.15 g/g). Onset (T_o), midpoint (T_m), and conclusion (T_c) gelatinization temperatures ranged between 63 to 79 °C. Peak, breakdown and setback viscosity were higher in *D. dumentorum* than *C. bicolor*. Moisture sorption behavior indicated increased hygroscopy with exposure time. The starches had high antinutrient levels, with *D. dumentorum* having higher levels of HCN and oxalates. In addition, infrared (IR) spectra of both starches were similar. Taken together, these properties suggest the suitability of these starches for non-food applications due to their high antinutrient contents.

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1. Introduction

Starch is the most abundant carbohydrate found naturally. Produced and stored in tissues of green plants, it is also the most common carbohydrate in human diet. Starchy tubers, root crops and cereals are important food sources in Nigeria and other parts of the world [1].

Starch finds wide application in the food and non-food industries because of their varying physicochemical and functional properties. Numerous studies exist on starches from cereals such as maize, wheat, millet, rice and root crops such as cassava, potato, yam, etc. for food and industrial purposes [2-9]. In Nigeria, these cereals and root crops are staple foods; this places huge constraints on the utilization of their starches for industrial purposes, hence the need to explore the properties of other starch- rich biomass becomes imperative.

Wild *C. bicolor* (family Araceae) and wild *D. dumentorum* (family Dioscoreaceae) are two underutilized, inedible root tubers found abundantly in the wild in the south and south east regions of Nigeria. *C. bicolor* is used as an ornamental plant in some areas, the leaves and rhizomes are used medicinally as topical application for boils, wounds and ulcers. It is used as purgatives and in the treatment of convulsion [10]. The potential of its flour for bio-ethanol production using indigenous fungal isolates has been reported [11]. Wild *D. dumentorum* is not eaten in Nigeria, but is cultivated and consumed in Ghana, Cameroun and other West African countries. Its flour is rich in protein (9.6%) with fairly balanced essential amino acid content and has a chemical score of 0.94. The starch granules have been reported to have a polygonal or spherical shape (<10 μm) [12]. Also, the nutritional quality of its flour for growing rat, tuber hardening phenomenon as well as changes in its antinutritional factors after harvest have been studied [12,13]. Furthermore, the use of starches from *C. bicolor* and *D. dumentorum* as dual purpose polymer additive [14] and their suitability for the synthesis of biodegradable starch plastics have been documented [8].

Inspite of their abundance, little information exist on the properties of its starches. The present study was therefore carried out to evaluate the physicochemical, functional and antinutritional properties of starches extracted from wild *C. bicolor* and *D. dumentorum*. This information could be used to access the suitability of starches from these plants for industrial applications.

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Table 1. Physicochemical properties of starches from *C. bicolor and D. dumentorum*.

Variable *	*C. bicolor*	*D. dumentorum*
Starch yield (%)	66.18±0.37	68.13±0.25
Moisture content (%)	7.65± 0.04	10.29±0.04
pH (20% slurry)	4.38± 0.05	4.09±0.04
Particle size (μm)	2-5	1-5
Ash content (%)	2.85±0.03	2.31±0.04
Crude lipid (%)	0.12±0.01	0.15±0.02
Crude protein (%)	0.11±0.01	0.13±0.01
Crude fibre (%)	2.85±0.12	3.39±0.21
Carbohydrate content (%)	83.57±7.11	82.62±8.22
Amylose content (%)	17.68±0.11	14.93±0.14
Amylopectin (%)	82.33±0.36	85.08±0.23

* Mean of triplicate determinations.

2. Experimental

2.1. Sample collection

Wild species of cocoyam (*C. bicolor*) and three leaf yam (*D. dumentorum*) were harvested from the wild within Uyo Local Government Area of Akwa Ibom State, Nigeria. The roots were thoroughly washed, peeled, sliced, grated and dried to homogenous flour.

2.2. Starch extraction

The modified method of Adikwu [15] was used. Briefly, 80 g of each sample was soaked in distilled water for 30 mins and filtered using a cheese cloth. This process was repeated four times and the filtrates combined, and then allowed to settle for 6 hrs. On complete sedimentation, the supernatant was discarded to obtain the starch, which was soaked in distilled water containing 0.1% Na_2SO_3 (*w*:*v*) for 24 hrs. After this, the starch was re-extracted with 70 mL of 0.1 M NaOH for 24 hrs and 70 mL of 0.1 M H_2SO_4 for 12 hrs. The obtained starch was dried at 60 °C for 12 hrs, weighed and stored in an air-tight container.

The percentage starch in each sample was determined using the Equation 1.

$$\%\ \text{Starch yield} = \frac{\text{Weight of dry starch}}{\text{Weight of dry flour}} \times 100 \quad (1)$$

2.3. Proximate analysis

Crude fibre, lipid, protein, carbohydrate and ash content of the isolated starches were determined by The Official Methods of Analysis of AOAC International methods [16].

2.4. pH determination

An electronic pH meter which has been standardized using buffer solutions 4 and 9, respectively, was used to determine the pH of the isolated starches (20% slurry).

2.5. Determination of particle size

This was determined using an Olympus laboratory microscope equipped with a graticule and camera. 70% of iodine solution in alcohol was added to 1% starch solution of each sample and placed in a glass slide with a glass cover slip and viewed under the microscope. A 40× objective lens and a 10× eyepiece were employed. The particle size of the starch grains were recorded with the help of the microscope graticule [17].

2.6. Determination of amylose and amylopectin content

The modified method of Mojzoobi *et al.* [18] was used to determine the amylose and amylopectin content of the isolated starches.

2.7. Antinutrient determination

Oxalate content of the starches was determined using the method of Ritter and Savage [19]. Tannins were evaluated using the method of Pearson [20]. Phytate was evaluated using the method of Nkama and Gbenyi [21]. Hydrogen cyanide was determined by the AOAC method [16].

2.8. Functional, pasting and thermal properties

Starch dispersibility was determined by the method described by Kulkarni, *et al.* [22]. Swelling power and solubility was evaluated at a concentration of 2% (*w*:*v*) at 90 °C in accordance with Nwokocha and Williams [23]. The centrifugal method of Beuchat [24] was used to determine the oil and water absorption capacity of the isolated starch samples. To evaluate the pasting properties of the samples (6%, *w*/*w*), a rapid viscoanalyser (Newport Scientific, RVA 4, Australia) was used. The pasting temperature (P_{temp}), peak viscosity (PV), trough viscosity (TV), breakdown viscosity (BV), final viscosity (FV) and setback viscosity (SV) were recorded in accordance with the method of Sandhu and Singh [25]. Gelatinization temperature of the samples was determined using differential scanning calorimeter (DSC) (Perkin-Elmer DSC-7, Norwalk, CT). The enthalpy change of gelatinization (ΔH_{gel}), onset (T_o), peak (T_p) and conclusion (T_c) temperatures of the gel were recorded [4].

2.9. IR spectra

IR spectra of the samples were carried out using an IR spectrometer (Shanghai S410, NIR Infrared).

3. Results and discussion

3.1. Physico-chemical properties

Wild *C. bicolor* and *D. dumentorum* grows abundantly in the south east and south south regions of Nigeria. Since they are not consumed, they represent an underutilised biomass that could be exploited for industrial purposes. As presented in Table 1, *D. dumentorum* had slightly higher starch yield (68.13±0.25%) than *C. bicolor* (66.18±0.37%). These were higher than *Manihot esculenta* (cassava), *Dioscorea hispida* and *Ipeoma batatas* (sweet potato) [2] but lower than reports by Alobi *et al.* [8]. These results suggest that *C. bicolor* and *D. dumentorum* flours are rich sources of starch that could be utilized industrially. Moisture content was lower in *C. bicolor* (7.65±0.04) than *D. dumentorum* (10.29±0.04); however, both were lower than the recommended safe storage range of <13 %, suggesting their suitability as commercial starch [18,26]. The pH of the starch slurry (20%) showed they are acidic (4.09±0.04-4.38±0.05) and was lower than *D. hispida* (4.48±0.03), *M. esculenta* (5.55±0.01), *Solanum tuberosum* (6.22±0.00), but higher than *D. pyrifolia* (3.43±0.05).

Table 2. Functional, pasting and thermal properties of starches from *C. bicolor and D. dumentorum*.

Variable*	*C. bicolor*	*D. dumentorum*
Functional properties		
Dispersibility (%)	53.49±0.01	64.85±0.07
Swelling power (g/g)	78.50±0.05	53.20±0.28
Solubility (%)	41.60±0.50	73.99±0.93
Water absorption capacity (g/g)	2.05±0.01	1.06±0.01
Oil absorption capacity (g/g)	1.56±0.01	2.15±0.01
Gelatinization characteristics		
T_o (°C)	63±0.4	69±0.3
T_m (°C)	67±0.4	76±0.4
T_c (°C)	75±0.5	79±0.4
ΔH_{gel} (J/g)	10.07	12.14
R	12	10
Pasting properties		
P_{temp} (°C)	69±0.3	75±0.4
PV (cP)	3033±48.1	3214±50.3
TV (cP)	1478±36.5	1782±27.5
BV (cP)	987±23.4	1135±30.1
SV (cP)	1395±44.9	1441±21.9
FV (cP)	2736±57.3	3574±44.7

* Mean of triplicate determinations; T_o = Onset gelatinisation temperature; T_m = Midpoint gelatinisation temperature; T_c = Conclusion gelatinisation temperature; R = Gelatinisation temperature range; ΔH_{gel} = Enthalpy of gelatinization; P_{temp} = Pasting temperature; PV= Peak viscosity; TV = Trough viscosity; BV = Breakdown viscosity; SV= Setback viscosity; FV = Final viscosity.

The starches had very small granule size, 1-5 µm [27]. *C. bicolor* and *D. dumentorum* starches had high ash contents (2.85±0.03 and 2.31±0.04%, respectively); these were higher than the recommended industry standard (0.5% ash) and reports by Alobi *et al*. [8]. This variation may be due to environmental factors, genetic mutation, processing and storage conditions. Lipid and protein were generally low, with *C. bicolor* (0.12±0.01 and 0.11±0.01%, respectively) having lower contents than *D. dumentorum* (0.15±0.02 and 0.13± 0.01%, respectively). These were lower than Chinese sweet potatoe, mung bean, *D. alata* and *D. esculenta* [2,28]. Low lipid and protein content of the isolated starches indicates high purity and quality. Also, high protein and lipid results in low clarity of starch paste and represses starch granule swelling [28,29]. Carbohydrate was similar in both starches (83.57±7.11 and 82.62±8.22% for *C. bicolor* and *D. dumentorum*, respectively) and did not differ significantly. However, they were lower than *D. pyrifolia* (92.73±0.48%), *D. alata* (86.81±1.33%), *Ipeoma batatas* (92.20±1.08%) but higher than *Solanum tuberosum* (80.22±0.00%) [2]. Crude fibre was slightly higher in *D. dumentorum* (3.39±0.21%) than *C. bicolor* (1.85±0.12%). Lower values have been reported for *Zea mays, Solanum tuberosum, Metroxylan sagu* and sweet potatoes, while higher values have been reported for mung beans [2,5].

3.2. Amylose and amylopectin content

Amylose and amylopectin content of the starches are given in Table 1. These are the two major components of starch granules and greatly affect the function of starch such as swelling, solubility, pasting, gelatinization, etc. Amylose has the proclivity for strong films and gels and to retrograde, while amylopectin forms softer gels and films when dispersed in water [9]. Generally, the starches had low amylose content, with *C. bicolor* being slightly higher (17.68±0.11%) than *D. dumentorum* (14.93±0.14%). Higher amylose contents have been reported for *D. pyrifolia* (44.47±1.86%), *D. opposite* (20.65±0.15%), *S. tuberosum* (26.90±0.08%), *L. meyenii* ecotypes (21.00-21.30±0.00%); similar values have been reported for *D. bulbifera* (17.61±0.13%), *D. nipponica* (17.67± 0.33%), while lower value is reported for *D. septemloba* (13.58±0.10%) [2,4]. The low amylose content observed in our starches suggests use in situations where soft gels/films are needed such as in the glue, detergent, plywood and textile industry.

3.3. Functional, pasting and thermal properties

Dispersibility of starch measures its degree of reconstitution in water. The higher its dispersibility, the better it reconstitutes in water [23]. *D. dumentorum* had a higher dispersibility (64.85±0.07%) than *C. bicolor* (53.49±0.01%) as given in Table 2. Higher dispersibility value has been reported for rice (87.01%), cassava (84.00%) and potato (86.00%) starch [30,31], while a lower value has been reported for breadfruit (40.67%) starch [32].

Swelling power and water solubility index provides evidence of the magnitude of interaction between starch chains with both the amorphous and crystalline domains [33], and gives a measure of the hydration status of starch molecules. *C. bicolor* starch had a higher swelling power (78.50±0.05 g/g) and lower solubility (41.60±0.05%) than *D. dumentorum* (53.20±0.28 g/g and 73.99±0.93%, respectively). Lower swelling power has been reported for *D. bulbifera*, *D. alata, D pyrifolia, D opposite* and cereals [3]. The extent of cross-bonding within the granules and the presence of lipids or phosphates affect the swelling and solubility profiles of starches. The high swelling power and solubility index exhibited by these starches may be attributed to its low amylose content and low lipid content as high amylose content and strong intermolecular bonds reduce swelling [5]. These suggest that these starches may find application in adhesives, pastes and glues in the non-food industries [34].

Water absorption capacity of the starches varied between 1.06±0.00 g/g and 2.05±0.01 g/g, with *C. bicolor* having a higher capacity than *D. dumentorum* (Table 2). The water absorption capacity of *C. bicolor* was higher than *D. opposite* (1.41±0.01 g/g), *D. alata* (1.56±0.11 g/g), but lower than *D. pyrifolia* (2.45±0.23 g/g), *D. bulbifera* (2.53±0.02 g/g) and *D. septemloba* (5.57±0.03 g/g). Water absorption capacity of *C. bicolor* and *D. dumentorum* starches is affected by the extent of hydrogen bonding between water molecules and starch hydroxyl groups as well as loose association between amylose and amylopectin molecules in starch granules [2,3].

Oil absorption capacity was higher in *D. dumentorum* (2.15±0.01 g/g) than *C. bicolor* (1.56±0.01 g/g). The oil absorption capacity of our starches was higher than rice and corn starches (1.09±0.03 to 1.1±0.03 g/g and 0.08±0.08 to 0.85± 0.07 g/g, respectively) grown in Indian temperate climate as well as tartary buckwheat starch (92.48±12.19 g/g) [6,34].

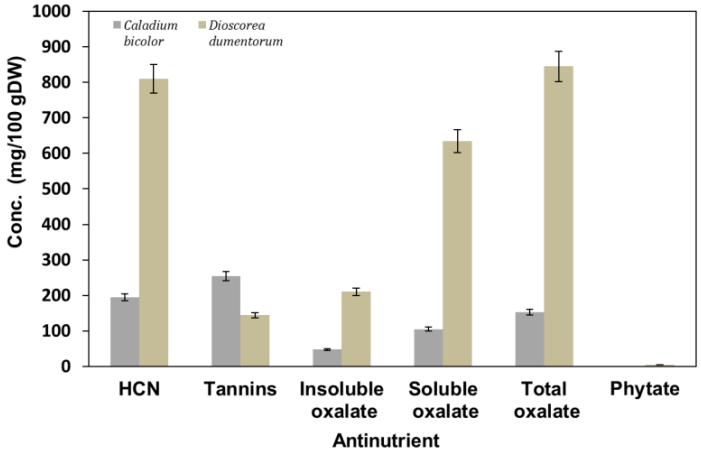

Figure 1. Antinutrient composition of starches from *Caladium bicolor* and *Dioscorea dumentorum.*

These result indicate that our starches can effectively absorb oil arising from the interaction between non polar amino acid side chain and hydrocarbon lipid chains within the starch granules, thereby enhancing mouth feel and flavour retention [6,34]. The starches formed odorless gels with similar viscosity, although *D. dumentorum* was slightly higher (1.48±0.01%) than *C. bicolor* (1.29±0.01%).

Gelatinization temperatures (onset T_o, midpoint T_m and conclusion T_c), enthalpy change of gelatinization (ΔH_{gel}) and gelatinization range (R) for the starches are given in Table 2. *D. dumentorum* had a higher gelatinization temperature (T_o = 69±0.3 °C, T_m = 76±0.4 °C, T_c = 79±0.4 °C) and enthalpy of gelatinization (ΔH_{gel}=12.14 J/g) than *C. bicolor* (T_o = 64±0.3 °C, T_m = 67±0.4 °C, T_c = 75±0.4 °C; ΔH_{gel} = 10.07 J/g), but with a shorter range (R = 10). Higher gelatinization temperatures have been reported for *D. opposite*, *D. pyrofolia*, *D. alata* [2], while lower gelatinization temperatures have been reported for maca starch [4] mung bean and potato [2]. According to Jiang *et al.* [3] gelatinization of starch is affected by the molecular arrangement of the crystalline region bearing the amylopectin chains and not the amylose-amylopectin ratio. In addition, variables such as amylose content, complexes formed by interaction between lipids and amylose chains, size and shape of the starch granules as well as the distribution of the amylopectin chains also affect the gelatinization characteristics of starches.

D. dumentorum had higher pasting (3214±50.3 cP), trough (1782±27.5 cP), breakdown (1135±30.1 cP), final (3574±44.7 cP) and setback viscosity (1441±21.9 cP) than *C. bicolor*. In addition, a higher pasting temperature was observed for *D. dumentorum* (75±0.4 °C) than *C. bicolor* (69±0.3 °C) (Table 2). This variation may be attributed to the molecular structure of their amylopectin chain, amylose content as well as their granular architecture. Higher values have been reported for maca starch [4] while lower values have been reported for corn starch [25].

3.4. Antinutrient composition

Levels of antinutrients in the isolated starches are given in Figure 1. HCN was higher in *D. dumentorum* (810.40±2.96 mg/100 g) than *C. bicolor* (194.85±0.48 mg/100 g). Samuel *et al.* [36] reported 6.54 mg/kg for tapioca flour. Ndidi *et al.* [37] reported HCN level of 224.03±2.54 mg/kg for African yam bean (*Sphenostylis stenocarpa*) seeds. Our results indicate high level of HCN in the starches above permissible levels. High HCN is capable of inhibiting cytochrome C oxidase, which can lead to cytotoxic hypoxia [38]. The high level of HCN may be due to the fact that during starch isolation, heat treatment was not involved, as antinutrients are heat labile and deactivated during processing protocols requiring heat treatment [39]. Tannins was higher in *C. bicolor* (254.97±0.42 mg/100 g) than *D. dumentorum* (144.71±0.06 mg/100 g). In comparison with other works, lower tannin levels are reported for edible trifoliate yam (22.00±0.00 mg catechin equivalent /100 g), tapioca (0.01%), cocoyam (0.05 g/100 g) and yellow yam (0.01±0.00 mg/100 g) [12,35,36,40]. However, our results were within permissible limits [37]. High oxalate levels were found in the starches. Total oxalate was higher in *D. dumentorum* (844.10±14.57 mg/100 g) than *C. bicolor* (153.16±18.15 mg/100 g). *D. dumentorum* also had higher levels of soluble and insoluble oxalates than *C. bicolor* (Figure 1). Samuel *et al.* [36] reported high oxalate level of 0.6% for tapioca, while Medoua *et al.* [12] reported 502-512 mg/100 g for *D. dumentorum* from Cameroun. In addition, high oxalate levels (780 mg/100 g) have been reported for taro corms [41]. High oxalate levels in diet interfere with mineral absorption and have been implicated in kidney stones [42]. Phytate levels were low and within permissible limits. It ranged from 0.24±0.06 mg/100 g to 4.66±0.05 mg/100 g, with *D. dumentorum* having the higher level. Higher values have been reported for tubers, legumes and cereals [12,35-37], while phytate level of 0.05±0.00 mg/100 g has been reported for *Dioscorea cayenensis* [39]. Phytates are known to bind mineral nutrients in the digestive tract leading to mineral deficiency in the body [43]. Overall, the starches had comparatively high levels of HCN, tannins and oxalates; suggesting their suitability for non-food applications.

3.5. Moisture sorption behavior

Moisture sorption behavior of *C. bicolor* and *D. dumentorum* is given in Figure 2. Result showed increased hygroscopy with exposure time. After 24 hrs, *C. bicolor* and *D. dumentorum* sorped 0.420 and 0.62 g of moisture, respectively. However, this decreased with increasing exposure time. At 120 hrs, moisture sorped was 0.016 and 0.017 g for *C. bicolor* and *D. dumentorum*, respectively; this equilibrated to 0.002 g for both samples at 192 hrs. *D. dumentorum* sorped more moisture between 24 to 120 hrs, while *C. bicolor* sorped more moisture after 120 hrs, until it equilibrated at 192 hrs. Our result is in agreement with reports for maize starch at 33% relative humidity for 186 hrs [35]. This result suggests that under atmospheric conditions, our starches are sensitive to atmospheric moisture.

Table 3. Infrared frequencies (cm^{-1}) of starches from *C. bicolor and D. dumentorum.*

C. bicolor	Assignment	*D. dumentorum*	Assignment
3378	O-H stretch	3282	O-H stretch
1702	C=O stretch	2927	C-H stretch
1626	Adsorbed water	1640	Adsorbed water
1149	C-O stretch	1422	C-O-H bend
1034		1355	C-H out-of-plane
865	CH, CH_2 deform.	1202	
669		1149	C-O stretch
576		913	
		860	CH, CH_2 deform.
		766	

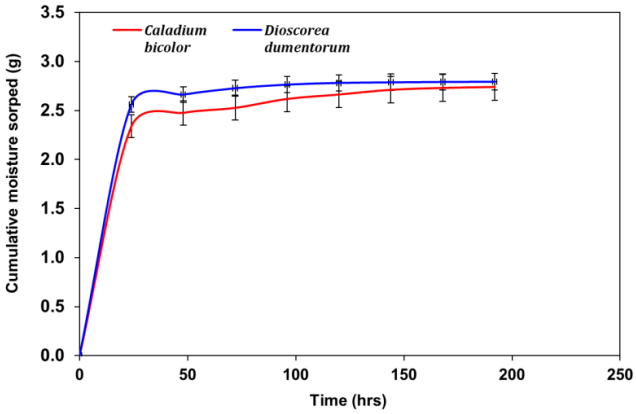

Figure 2. Moisture sorption behaviour of starches from *Caladium bicolor* and *Dioscorea dumentorum.*

3.6. IR spectroscopy

The observed bands for *C. bicolor* and *D. dumentorum* are given in Table 3. In the O-H stretching region, peaks were observed at 3282 cm^{-1} (broad) for *D. dumentorum* and 3378 cm^{-1} for *C. bicolor*. Because the C-H stretching vibrations occur in a very narrow region, overlap of bands may occur. C-H stretching frequency was observed at 2927 cm^{-1} for *D. dumentorum*, but absent in *C. bicolor*. This situation may be explained in terms of band interferences or overlap as well as the effect of α-D-glucose on the α-1,4-backbone and α-1,6-branch structure of the amylopectin moiety, causing a shift in the C-H stretching vibration of *C. bicolor*. Santha *et al.* [44] reported similar observation for the IR spectra of sweet potato and cassava starch. In the mid-range region, the bands at 1640 and 1626 cm^{-1} for *D. dumentorum* and *C. bicolor* respectively, may be attributed to adsorbed water [44], suggesting vibrations of adsorbed water molecules in the non -crystalline portion of the starches, while the band at 1702 cm^{-1} observed in *C. bicolor* may be due to minor component (lipids or protein) present in the sample. In the fingerprint region, peaks between 1100 and 1150 cm^{-1} may be attributed to C-O, C-C and C-O-H stretching, while peaks between 1100 and 900 cm^{-1} may be attributed to C-O-H bending modes. Peaks at 860 and 865 cm^{-1} in *D. dumentorum* and *C. bicolor* correspond to CH and CH_2 deformation modes. Similar results have been reported for potato starch [45]. However, it should be noted that in the IR spectra of starches, absorbance bands overlap and may be poorly resolved, hence it becomes difficult to assign bands unambiguously [46].

4. Conclusion

Starch isolated from *C. bicolor* and *D. dumentorum* had variable physicochemical, functional, thermal, pasting and antinutrient properties. Moisture and amylopectin content was higher in *D. dumentorum* than *C. bicolor*, while carbohydrate content was similar. In addition, both starches had good swelling power, dispersibilty, oil and water absorption capacity as well as thermal and pasting properties. However, their high antinutrient content possesses a challenge to its use in the food industry. This suggests that these starches may find great use in paper making, adhesives, textile and other non-food applications.

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Disclosure statement

Conflict of interests: The authors declare that they have no conflict of interest.
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