COMPRESSIONAL CHARACTERISTICS AND TABLETING PROPERTIES OF STARCHES OBTAINED FROM FOUR *DIOSCOREA* SPECIES

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Abstract

The compressional characteristics and tableting properties of starches from four yam species namely *Dioscorea dumetorum* Pax (Bitter); *Dioscorea oppositifolia* L (Chinese); *Dioscorea alata* L.DIAL2 (Water) and *Dioscorea rotundata* Poir (White) were investigated in comparison with corn starch. The physicochemical properties of the starches were evaluated using established methods while the compressional characteristics were analyzed using density measurements, and the Heckel and Kawakita equations. The properties of the tablets were assessed using Tensile strength (T), Brittle Fracture Index (BFI), Friability (F) and Disintegration Time (DT). The physicochemical properties of the starches varied considerably among the various species. The ranking for the mean yield pressure (P_y) obtained from Heckel plots was Chinese > Bitter>Corn > White>Water while the ranking was the reverse for another pressure term, P_k, obtained from Kawakita plots. The ranking for T was Chinese > Bitter>Corn > White>Water. The T values were inversely related to P_k values. The ranking of DT was Bitter>Chinese>Corn>White> Water, while the ranking was reverse for BFI and F. Water and White yam starch tablets did not conform to the Pharmacopoeia requirements on friability (≤1%) while all the starch tablets except Bitter yam starch conformed to the requirements on disintegration (≤ 15minutes). Thus, the starches could be useful as excipients in tablet formulations.

Rezumat

Lucrarea prezintă un studiu comparativ privind caracteristicile de comprimare şi tabletare a patru tipuri de amidon provenind de la specii de *Dioscorea* (*Dioscorea dumetorum, Dioscorea oppositifolia, Dioscorea alata, Dioscorea rotundata*), în raport cu amidonul de porumb. Proprietățile fizico-chimice ale celor patru tipuri de amidon au fost cercetate prin metode oficinale în Farmacopeea Britanică. Caracteristicile de comprimare au fost studiate utilizând determinări densitometrice şi ecuațiile Heckel şi Kawakita.

Keywords: Yam Starch, tablets, compressional properties

Introduction

Starches are widely available, naturally occurring carbohydrate, reserves found in almost all organs of plants, most especially in roots, rhizomes, fruits and seeds. As part of the effort to reach harmonization of pharmaceutical excipients, the Joint Conference on Excipients identified starches as one of its top 10 excipients [27]. Starches have been used as multifunctional excipients in tablet formulations due to their relative inertness, abundance, cheapness and suitable physicochemical properties [3, 26]. Starches are used as diluents, glidants, binders and disintegrants and many authors have studied starches obtained from different botanical sources as excipients in tablet formulations [12, 3, 2, 20]

Yams, which are annual or perennial tuber-bearing and climbing plants, belong to the genus *Dioscorea*, a genus of over 600 species of flowering plants in the family Dioscoreaceae [5]. In many parts of Africa and South East Asia, yam is a primary agricultural commodity, classified as the third most important tropical root crop after cassava and potatoes. The high starch content of yam tubers (70 to 80% dry weight) has made them a potential source of starch that could be explored commercially [24, 8].

In recent years, attention has been focused on edible varieties of *Dioscorea* as potential source of native starch that could find some applications in pharmaceutical industries. A major factor limiting the use of these starches in pharmaceutical formulations is the lack of adequate information on their physicochemical, fundamental and derived properties. Studies have been performed on the binding and disintegrant properties of yam starch obtained from *D. rotundata* [18], packing and cohesive properties of *D. rotundata* [14] and the film forming properties of starches obtained from *D. alata* [7]. Although the physicochemical, thermal, morphological and material properties of the starches from four *Dioscorea* species has been investigated [21], no work has been done on the compressional and tablet properties of these starches with a view to determining the relative usefulness of these species in term of known applications.

Thus in the present study, the compressional and tableting properties of starches obtained from four species of yam namely *Dioscorea dumetorum* Pax (Bitter yam); *Dioscorea oppositifolia* L (Chinese yam); *Dioscorea alata* L. DIAL2 (Water yam) and *Dioscorea rotundata* Poir (White yam) have been investigated in comparison with the official corn starch. The compressional characteristics were analyzed using the Heckel and Kawakita equations [9, 15] while the tablet properties were determined using their tensile strength (T), Brittle Fracture Index (BFI) [6, 10], friability and disintegration time (DT).

Materials and Methods

Materials

The materials used were cornstarch BP (BDH Chemicals Ltd, Poole, U.K.), fresh tubers of four yam species namely *Dioscorea dumetorum* Pax

(Bitter); *Dioscorea oppositifolia* L (Chinese); *Dioscorea alata* L.DIAL2 (Water) and *Dioscorea rotundata* Poir (White) obtained from local farmers in Ibadan, Nigeria and authenticated. The starches were isolated from the tubers according to established procedures [33]. In the following text only the abbreviations Bitter, Chinese, Water and White will be used for the yam starches.

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 2kV.

Particle size and shape

The particle size distribution of each starch was determined by optical microscopy (Leitz, laborlux II, Germany) on 300 particles. This was used to determine the mean projected diameter (d) and the particle shape. The shape coefficient, α, for all the starches was calculated from the expression [17]:

$$\alpha = \rho_s \, Sw \, de \; + \; N \qquad (1)$$

where N is the elongation ratio (= length / breadth). The Heywood diameter, de, is obtained from the expression $[(4 \times 0.77 \times L \times B) / \pi]^{1/2}$μm. L and B are the mean length and breadth of the particles respectively.

The surface area of the starches, Sw (m^2/g), was calculated using the expression [30]:

$$Sw = \alpha_{sv.t} \, Sv / \rho_s \qquad (2)$$

where $\alpha_{sv.t}$ is the surface-volume sieve shape factor, which was taken as 6, i.e the shape factor for a sphere, Sv is the geometric surface area of the starches in m^{-2} obtained from sieve analysis and ρ_s is the particle density in gm^{-3}.

Angle of repose

The angle of repose was calculated from an open ended cylinder of diameter 2.8cm placed on a base of similar diameter. 30g of starch powder was allowed to flow through a funnel, under the force of gravity to form a conical heap. The angle of repose was calculated as a mean of four determinations as:

$$\tan \theta = h / r \qquad (3)$$

where h is the height of the cone and r is the radius of the base of the cone.

Determination of density parameters

The apparent particle density of all equilibrated starches was determined by Helium pycnometry (Accupyc 1330; Micromeritics, Norcross, GA, USA) in triplicate. The bulk density of each starch powder at zero pressure (loose density) was determined by pouring the powder at an angle of 45° through a funnel into a glass measuring cylinder with a diameter of 21mm and a volume of 50ml [22, 12]. Determinations were performed in quadruplicate. The relative density, D_o, of each starch powder was obtained from the ratio of its loose density to its particle density.

The Hausner`s ratio determined as the ratio of the initial bulk volume to the tapped volume, was obtained by applying 100 taps to 30g of starch in a graduated cylinder at a standardized rate of 38 taps per minute (British Standard, 1970).

Preparation of starch tablets

Quantities (600mg) of starch powder was compressed for 30 seconds into tablets with predetermined loads on a Carver hydraulic press (model C, Carver inc. Menomonee Falls, WI) using a 10.5mm die and flat-faced punches lubricated with a 1% dispersion of magnesium stearate in acetone before each compression. Tablets with a hole (1.54mm diameter) at their center were made using an upper punch with a hole through the center and a lower punch fitted with a pin [12]. After ejection, the tablets were stored over silica gel for 24hours to allow for elastic recovery and hardening. The weights (w) and dimensions of the tablets were determined within ± 1mg and 0.01mm, respectively. The relative density of the tablets (D) was calculated using the equation:

$$D = w / V_t . \rho_s \qquad (4)$$

where V_t is the volume (cm^3) of the tablet, including the hole when present.

Compaction Data Analysis

The compression equations of Heckel and Kawakita were used to assess the compaction properties of the starches [18]. The Heckel equation is widely used for relating the relative density, D, of a powder bed during compression to the applied pressure, P. It is written as:

$$\ln [1 / (1 - D)] = K P + A \qquad (5)$$

The slope of the straight line portion, K, is the reciprocal of the mean yield pressure, P_y, of the material. From the value of A the intercept, the relative density, D_A, can be calculated using the following equation [11]:

$$D_A = 1 - e^{-A} \qquad (6)$$

The relative density of the powder at the point when the applied pressure equals zero, D_o, is used to describe the initial rearrangement phase of densification as a result of die filling. The relative density D_B, describes the phase of rearrangement at low pressures and is the difference between D_A and D_O:

$$D_B = D_A - D_O \quad (7)$$

The Kawakita equation is used to study powder compression using the degree of volume reduction (C) and is written as:

$$C = (V_o - V_p) / V_o = a\,b\,P / (1 + b\,P) \quad (8)$$

The equation, in practice can be rearranged to give:

$$P / C = P / a + 1 / ab \quad (9)$$

where V_o is the initial bulk volume of the powder and V_p is the bulk volume after compression. The constant a is equal to the minimum porosity of the material before compression while the constant b is related to the plasticity of the material. The reciprocal of b gives a pressure term P_k which is the pressure required to reduce the powder bed by 50% [28, 16].

Tablet properties

The tensile strength of the normal tablets (T) and apparent tensile strength of the tablets containing a hole (T_o) were determined at room temperature by diametral compression (Fell and Newton, 1970) using a hardness tester (Ketan Scientific & Chemicals, Ahmedebad, India) and by applying the equation:

$$T = 2F/\pi dt \quad (10)$$

where T (or T_o) is the tensile strength of the tablet (MNm^{-2}), F is the load (MN) required to cause fracture, d is the tablet diameter (m) and t is the tablet thickness (m). All measurements were performed in quadruplicates.

The brittle fracture index (BFI) values of the tablets were calculated using the equation:

$$BFI = 0.5[(T/ T_o) - 1] \quad (11)$$

The friability percent of the tablets was determined using a friabilator (Veego Scientific devices, Mumbai, Maharashtra, India) operated at 25 rpm for 4 minutes. Determinations were performed in quadruplicates.

The disintegration time of the tablets was determined in distilled water at 37±0.5 ^oC using a disintegration tester (Veego Scientific devices, Mumbai, Maharashtra, India). Determinations were performed in quadruplicates.

Results and discussion

The particle shape has been shown to influence the compaction characteristics since it affects the packing behavior of the starches [18]. This is because there is a tendency for particle rearrangement to occur in the initial stages of the compaction process [32]. The scanning electron micrographs (SEM) of the starches are shown in Fig 1.

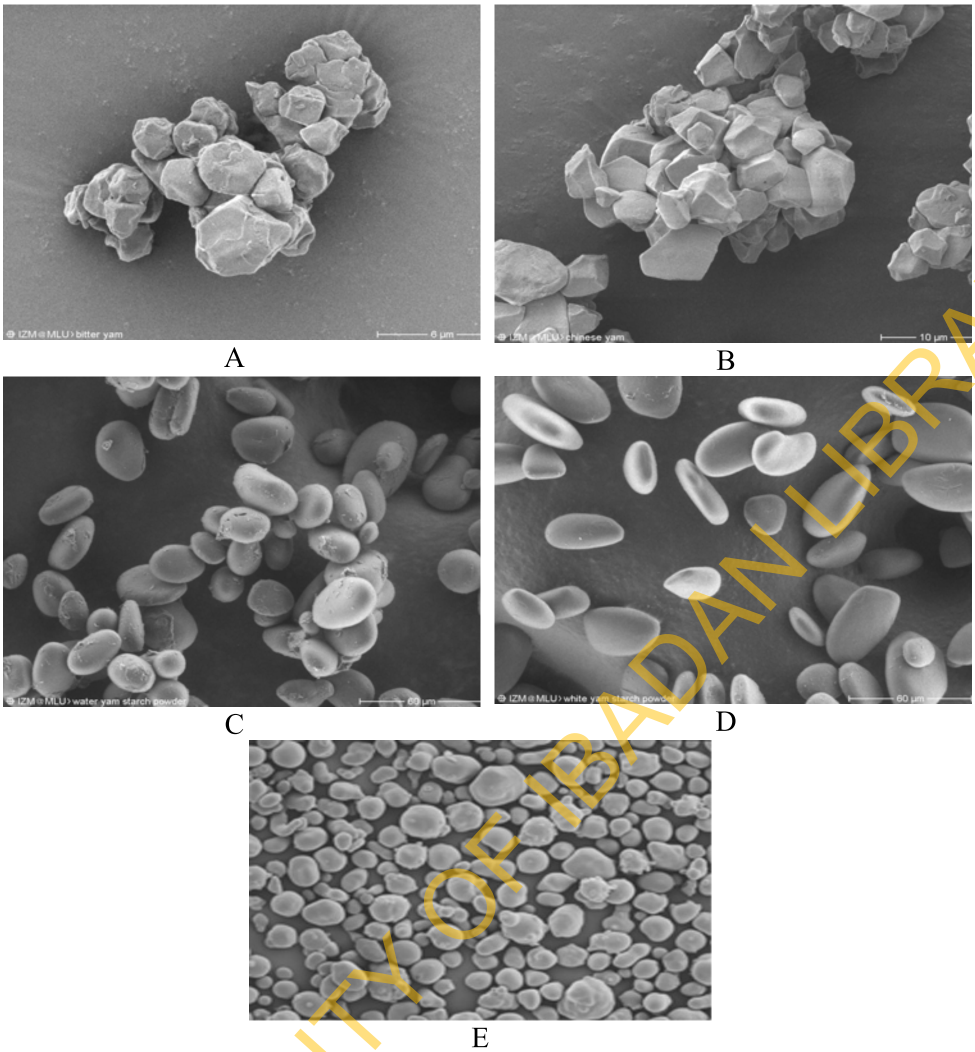

Figure 1
SEM of *Dioscorea* and corn starches: A, Bitter; B, Chinese; C, Water; D, White; E, Corn

The micrographs show that the shape of the *Dioscorea* starches varied considerably among the various species. Chinese and Bitter had granules that are polygonal in shape and existed as aggregates while White and Water had oval and ovoid shapes respectively. These observed shapes were consistent with those reported in literature [1]. Corn starch on the other hand had granules that were angular or polyhedral in shape.

The physical and geometric properties of the starches are presented in Table I. The ranking of the particle size, Heywood diameter and the shape coefficient was White>Water>Corn>Chinese>Bitter. White and Water had granule sizes larger than those of Corn while Chinese and Bitter exist as aggregates with smaller granule sizes. The shape coefficient serves as a proportionality constant between particle size and surface area [25]. Thus, Bitter which had the lowest shape coefficient was smaller and as the shapes became more angular as observed with the oval shape of White, the shape coefficient increased [22].

Table I

Physical and geometric properties of the various starches

Starch	Geometric shape	Mean particle diameter (µm)	d_e (µm)	R (µm)	S_w (m^2/g)	α
Bitter	Polygonal	3.45	2.62	1.70	0.016	0.07
Chinese	Polygonal	7.40	6.48	1.28	0.010	0.09
Water	Oval	29.00	24.44	1.38	0.042	1.53
White	Ovoid	44.00	36.05	1.46	0.043	2.31
Corn	Polyhedral, angular	14.60	7.05	1.08	0.015	0.16

d_e= Heywood diameter (µm); R= radius (µm); S_w= the surface area of the starches (m^2/g); α= the shape coefficient

The packing and cohesive properties of starches influence the various aspects of powder processing such as milling, blending, flow from hoppers, compression and packing into capsule shells or containers. The packing and cohesive properties of starches depend to a large extent on the particle size distribution and shape [14]. The angle of repose θ could be used as a qualitative measure of the cohesiveness or the tendency of powdered or granulated materials to flow, for instance, from hoppers through the feed frame into tableting machines. Such uniformity of flow will minimize weight variations in tablets produced [31]. Angles of 30◦ or below are usually indicative of free flowing materials while an angle of 40◦ or above indicates a poor flow. The angle of repose is affected by the particle size distribution and it usually increases with a decrease in particle size [23]. The angles of repose for the various starches are presented in Table II. The ranking of angle of repose was: Bitter> Chinese>Corn> Water>White. The ranking is inversely related to the ranking of the particle size of the starches. The angles of repose of all starches were above the range of 40◦, indicating they all have poor flow properties. It has been shown that cohesiveness is usually inversely related to the particle diameter of materials [29]. In general, the smaller the particle size and shape factor, the more cohesive the particles.

Table II

Material properties of the starches (Mean ± SD, n = 4)

Starch	Particle density (g/cm^3)	Bulk density (g/cm^3)	Hausner ratio	Angle of repose (˚)
Bitter	1.50±0 .01	0.45 ±0.00	1.64 ±0.02	63.60 ±1.42
Chinese	1.51±0.01	0.43 ±0.00	1.65 ±0.02	62.60 ±0.40
Water	1.47 ± 0.01	0.77 ± 0.00	1.10 ± 0.01	60.20 ± 0.95
White	1.48 ±0.01	0.76± 0.00	1.19± 0.02	61.04± 1.21
Corn	1.49±0.01	0.47± 0.00	1.44±0.01	61.80 ±0.38

The particle densities of the starches are presented in Table II. The ranking of the particle density was Chinese>Bitter > Corn>Water>White, while the ranking was reverse for the bulk density. 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. Higher bulk density is advantageous in tableting due to reduction in the fill volume of the die. The differences observed in the bulk density values could be due to the different particle shapes which affected the packing arrangement of the powder particles. The Hausner ratio, i.e. ratio of tapped to bulk density indicates the degree of densification which could occur during tableting with the higher values predicting significant densification of powders. The ranking of the Hausner ratio was Chinese> Bitter>Corn>White>Water.

The Heckel plots for the starches are presented in Fig 2. The values of mean yield pressure, P_y, were calculated from the region of plots showing the highest correlation coefficient for linearity of > 0.997 for all the starches (generally between 56.56-169.69 MNm^{-2}). The intercept, A, was determined from the extrapolation of this region. Values of P_y, D_o, D_A and D_B for all the starches are presented in Table III. The ranking of the values of D_o was Water>White>Corn>Bitter>Chinese. This implies that Water and White exhibited higher degree of initial packing in the die as a result of die filling. This could be attributed to the respective ovoid and oval shapes of their particles which would facilitate closer packing of the particles than the polygonal shapes of the particles of the other starches. Furthermore, the large particle sizes of Water and White particles implies that they had less electrostatic forces that could prevent the packing of particles in the precompression stage.

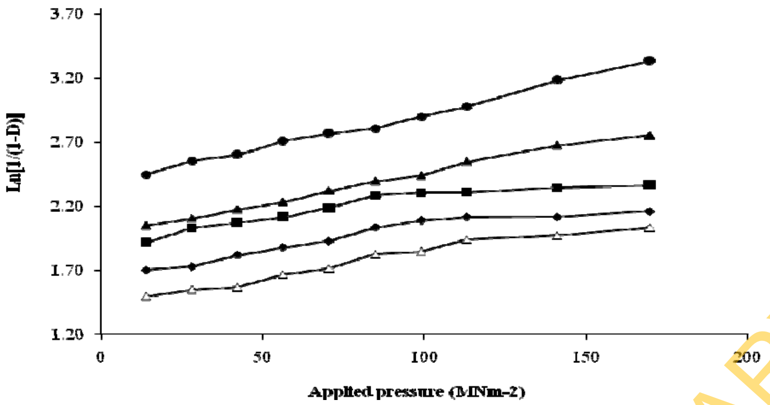

Figure 2
Heckel plots for tablets made from starches: ♦, Bitter; ■, Chinese; ●, Water; ▲, White; Δ, Corn

Table III
Parameters derived from density measurements and from the Heckel and Kawakita plots

Starch		Heckel Plots			Kawakita Plots	
	D_o	P_y (MNm^{-2})	D_A	D_B	D_I	P_k (MNm^{-2})
Bitter	0.302	303.03	0.811	0.509	0.336	1.451
Chinese	0.286	344.83	0.859	0.573	0.371	1.121
Water	0.523	178.57	0.907	0.384	0.622	3.679
White	0.512	208.33	0.862	0.350	0.533	3.618
Corn	0.316	243.90	0.761	0.445	0.355	2.496

D_0= the relative density (g/cm^3); P_y= the mean yield pressure; D_A= the relative density according to equation 6 in text; D_B= the relative density according to equation 7 in text; P_k= the pressure required to reduce the powder bed by 50%

The value of D_B represents the phase of rearrangement of the particles in the early stages of compression. D_B values tend to indicate the extent of fragmentation of particles or granules, although fragmentation can occur concurrently with plastic and elastic deformation of constituent particles. The ranking of the values of D_B was Chinese> Bitter> Corn> Water> White. Thus Chinese had the highest D_B values while White showed the lowest values.

The value of D_A represents the total degree of packing achieved at zero and low pressures. The ranking of D_A was Water> White> Bitter> Chinese> Corn. Thus, Water showed the highest D_A values while Corn showed the lowest values.

The mean yield pressure, P_y, is an inverse measure of the ability of the material to deform plastically under pressure. The ranking of P_y was Chinese>Bitter>Corn>White>Water. This shows that Water exhibited the fastest onset of plastic deformation during compression while Chinese exhibited the slowest.

The Kawakita plots for the starches are presented in Fig 3. A linear relationship was obtained at all compression pressures employed with correlation coefficient of 0.999 for all the starches. Values of *a* and *ab* were obtained from the slope and intercept respectively. Values of 1-*a* give the initial relative density of the starches, D_i, while P_k values were obtained from the reciprocal of values of *b*. The values of D_i and P_k are shown in Table III.

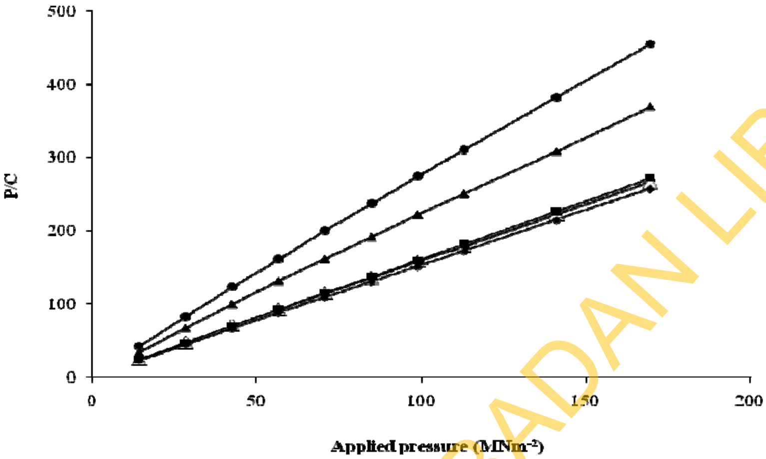

Figure 3
Kawakita plots for tablets made from starches: ♦, Bitter; ■, Chinese; ●, Water; ▲, White; Δ, Corn

The value of D_I is a measure of the packed initial relative density of the formulation with the application of small pressure or tapping [19]. The ranking of D_I was Water>White>Chinese >Corn>Bitter. The values of D_i for the starches were observed to be higher than the corresponding values of D_o. This results are in agreement with earlier findings where D_o provides a measure of the loose initial relative density while D_i provides a measure of the packed initial relative density of the material [19].

The values of P_k which is an inverse measure of the amount of plastic deformation occurring during the compression process [18] was Chinese< Bitter< Corn< White< Water. Thus, Chinese exhibited the highest amount of total plastic deformation while Water exhibited the lowest values. The ranking was seen to be in the reverse order as the Py values. It has been shown that while P_y relates to the onset of plastic deformation during compression while P_k relates to the amount of plastic deformation that occurs during the compression process [19]. Thus, Chinese which showed the slowest onset of plastic deformation showed the highest total amount of plastic deformation.

The results of the tensile tests on the paracetamol tablets fit the general equation:

$$\text{Log } T \text{ (or } T_o) = AD + B \qquad (12)$$

with a correlation coefficient > 0.991. A and B are constants which depend on the nature of starch involved and on the presence of a hole in the tablet. Values of T and BFI for the starches at relative density of 0.90, which is representative of commercial tablets, are presented in Table IV. The ranking of T for the starches was Chinese>Bitter>Corn> White >Water while the ranking was Water>White> Corn>Chinese>Bitter for BFI. Thus, Chinese and Bitter yam starches which showed higher T than corn starch also had lower BFI values than corn starch. Obviously, a low value of BFI is desired for the minimization of lamination and capping during tablet production. On the other hand, the desirable effect on tensile strength largely depends on the intended use of tablets [19]. Furthermore, there appears to be a relationship between the P_k values and the tensile strength of the tablets. Low P_k values of tablets have been shown to be responsible for high T values as higher total plastic deformation would lead to more contact points for interparticulate bonding [13, 19].

Table IV

Values of Tensile Strength (T), Brittle Fracture Index (BFI), Friability and Disintegration Time (DT) at relative density D = 0.90 for the tablets of *Dioscorea* and corn starches

Starch	T (MNm^{-2})	BFI	Friability %	DT (Min)
Bitter	1.40 ±0.17	0.11 ±0.04	0.06 ±0.00	29.33±0.58
Chinese	1.63 ±0.06	0.25 ±0.02	0.13 ±0.01	7.20 ±0.35
Water	0.56 ±0.17	0.48 ±0.34	1.80 ±0.10	0.29 ±0.03
White	0.60 ±0.02	0.47 ±0.05	1.38 ±0.03	0.42±0.01
Corn	0.71 ±0.01	0.43 ±0.13	0.43 ±0.01	0.50 ±0.02

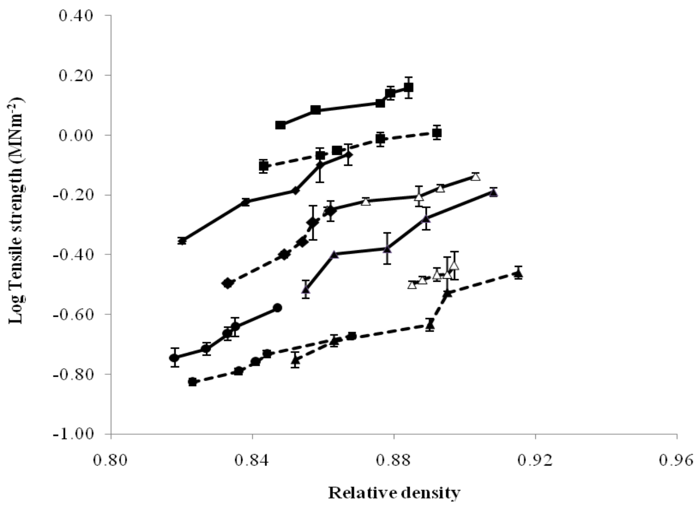

Figure 4
Log tensile strength versus relative density for tablets made from starches with hole (……) and without a hole (____) at their center : ♦, Bitter; ■, Chinese; ●, Water; ▲, White; Δ, Corn

The plots of friability versus relative density of the tablets are shown in Fig 5. The tablet friability decreased with relative density. Values of friability for the starches at relative density of 0.90, which is representative of commercial tablets, are presented in Table IV. The ranking of friability for the starch tablets was Water>White> Corn>Chinese>Bitter. The ranking is similar to that of BFI for the starches. Bitter and Chinese yam starch tablets conformed with the official pharmacopoeial requirements on friability by showing F values ≤ 1 % while Water and White yam starch tablets did not conform to the requirements on friability.

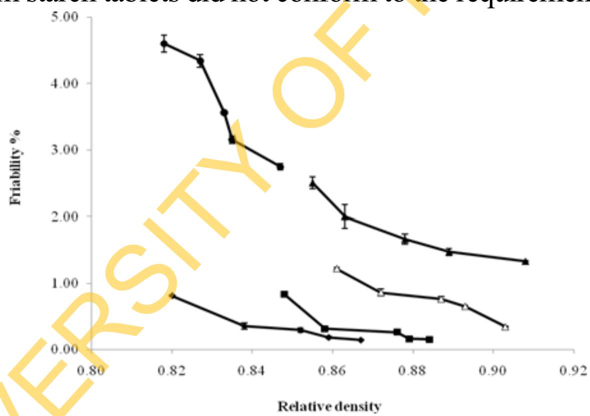

Figure 5
Friability versus relative density for tablets made from starches: ♦, Bitter; ■, Chinese; ●, Water; ▲, White; Δ, Corn

The plots of disintegration time (DT) versus relative density of the tablets are shown in Fig 6. Disintegration time increased with the increase in relative density in the rank order for Bitter>Chinese>Corn>White>Water tablets. However, all the starch tablets conformed to the Pharmacopoei requirements on disintegration by showing DT values ≤ 15minutes except Bitter.

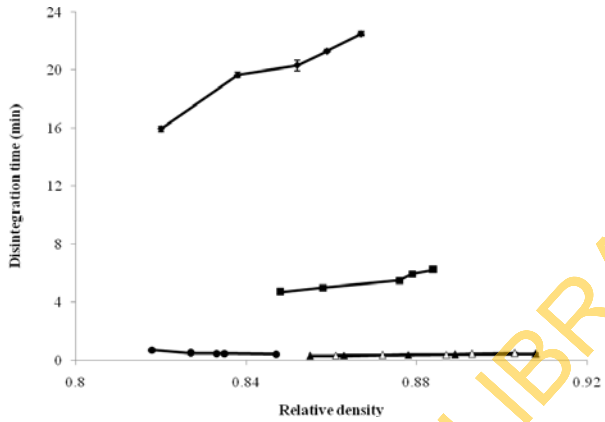

Figure 6
Disintegration time versus relative density for tablets made from starches: ♦, Bitter; ■, Chinese; ●, Water; ▲, White; Δ, Corn.

The results obtained have indicated that the starches from the four *Dioscorea* species vary considerably from one species to the other. The morphology and material properties of the starches varied considerably. The irregular shape of the particles of Bitter and Chinese yam starches resulted in higher cohesiveness of the powders which facilitated higher bond formation. An inverse relationship was observed between the P_k values and the tensile strength of the starches. The tensile strengths of Chinese and Bitter compacts were much higher than those of corn starch and the other *Dioscorea* starches. The tensile strength of the tablet is affected by the area of contact among particles which determines the strength of bonds produced among the particles. This resulted in tablets with higher mechanical strength and a higher ability to resist capping and lamination tendency of the starch tablets than corn starch.

Conclusion

The results of the study provide an insight into the material, compressional and tablet properties of the four *Dioscorea* starches in comparison with the official corn starch. The particle size, shape and specific surface area appear to play a significant role in the compressibility of the starches. The four *Dioscorea* starches generally exhibited poor flow properties and deform mainly by plastic flow. Water and White showed faster onset of plastic deformation than corn starch while Bitter and Chinese showed higher amounts of total plastic deformation. Bitter and Chinese starch tablets had higher tensile strengths than corn starch and longer disintegration times suggesting their potential use as binding agents in tablet formulations. On the other hand, Water and White yam starches produced tablets with lower tensile strength and shorter disintegration times than corn starch suggesting their potential use as disintegrants. Thus, the *Dioscorea* starches could be used as substitute for corn starch depending on the intended use of the formulation.

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