



# Material properties and compaction characteristics of natural and pregelatinized forms of four starches

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## ABSTRACT

Material properties and compaction characteristics of natural and pregelatinized forms of white trifoliolate (T.) yam, yellow trifoliolate (T.) yam, rice and official corn starches were studied. Physico-chemical properties and viscosity profiles of the starches were evaluated. Pregelatinized starches exhibited lower viscosity values than natural starches in the viscoamylography. The pregelatinized starches showed more densification than natural starches during compression and also had lower mean yield pressure,  $P_y$ . The ranking of  $P_y$  for the starches was; corn < white T. yam < yellow T. yam < rice. The values of  $P_k$ , an inverse measure of plasticity, were, however, lower for the natural starches with the ranking for the starches being white T. yam < yellow T. yam < corn < rice. Tablets produced from pregelatinized starches had lower values of tensile strength (T) and brittleness (BFI) than those made from natural starches. The ranking of T for the starches was generally white T. yam > corn > yellow T. yam > rice. The rankings of the BFI for the natural and pregelatinized starches were corn < yellow T. yam < white T. yam < rice; and corn < white T. yam < yellow T. yam < rice, respectively.

The material properties of the starches appear to show some limited correlation with their tablet properties. The experimental starches showed similar properties to official corn starch in many cases and could serve as suitable alternatives for particular purposes.

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## 1. Introduction

Starches are widely employed as multipurpose excipients in various solid dosage forms, especially as diluents, disintegrants, and binding agents in tablet formulations due to their suitable physico-chemical properties as well as their relative cheapness and inertness (Alebiowu & Itiola, 2001, 2002; Atichokudomchai & Varavinit, 2003). The versatility of starches implies a need to continue to develop new starch excipients with suitable properties to meet the special needs of drug formulators and the demands of novel formulations.

Pregelatinization of natural starches has been used to produce cold-water swellable forms with improved flowability (Alebiowu & Itiola, 2001, 2002; Visavarungroj & Remon, 1991). This process, which leads to irreversible granule swelling, loss of birefringence, and crystallinity, is usually done by heat treatment (Freitas, Paula, Feitosa, Rocha, & Seirakowski, 2004; Herman, Remon, & De-Vilder, 1989; Thomas & Atwell, 1999). Other methods such as solvent based processing, oxidation, hydrolysis and cross-linkage have also been used (Atichokudomchai & Varavinit, 2003; Thomas & Atwell, 1999).

*Dioscorea dumetorum* Pax, trifoliolate yam belongs to a large family of dioscoreaceae and it refers to both the edible and non-edible (wild) varieties (Nkala, Sibanda, Tomasik, & Palasinski, 1999). Peeled tubers of *D. dumetorum* is employed in the treatment of certain tropical diseases and some conditions such as infertility (Watt & Breyer-Brandwijk, 1962), but its tubers, as a source of starch, have attracted rather limited interest (Nkala et al., 1999).

On the other hand, milled rice grain (*Oryza sativa*, Linn) is made up of 85% starch with starch granule size of about 2–6  $\mu\text{m}$  (Ayernor, 1985). It appears however, that little attempt has been made to study the fundamental and compression properties of this particular starch with a view to determining its usefulness in tablet formulation and production. In addition, little attempt appears to have been made to investigate how the viscosity profiles (rheological properties) of starches can be useful in explaining their compressional characteristics.

In the present investigation, the physico-chemical, rheological and compression properties of natural and pregelatinized starches of rice and trifoliolate yam (the white and yellow types), as well as the mechanical properties of their tablets were studied in comparison with those of official corn starch BP. The compression properties were assessed using density measurements and Heckel and Kawakita equations (Heckel, 1961; Kawakita & Ludde, 1970/

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71) while tensile strength and brittle fracture index (BFI) were used to assess the mechanical properties of the starch tablets (Alebiowu & Itiola, 2002; Kawakita & Ludde, 1970/71; Lin & Cham, 1995).

Tensile strength has been used to measure the bond strength of tablets (Odeku & Itiola, 1998) and is calculated using the equation (Fell & Newton, 1970):

$$T = 2F/\pi dt \quad (1)$$

where  $F$  is the load (MN) required to cause fracture while  $d$  and  $t$  are tablet diameter (m) and thickness (m), respectively.

Brittle fracture index is a measure of the lamination tendency of tablets (Heistand, Wells, Poet, & Ochs, 1977). It is calculated using the equation:

$$\text{BFI} = 1/2(T/T_h - 1) \quad (2)$$

where  $T$  is the tensile strength of the tablets without a central hole and  $T_h$  is the apparent tensile strength of tablet having a hole, both measured at the same relative density. The hole acts as a built-in stress concentrator defect.

## 2. Experimental

### 2.1. Materials

The four natural starches used were corn starch BP (BDH chemicals, UK) and the starches of white trifoliate yam (white T. yam), yellow trifoliate yam (yellow T. yam) and rice prepared in the laboratory of the Department of Pharmaceutics and Industrial Pharmacy, University of Ibadan, Nigeria.

Tubers of white and yellow varieties of trifoliate yam (*D. dumetorum* Pax) were purchased from a local farm near Oyo town, Nigeria. The specimen tubers and shoots of the plants were authenticated in the herbarium department of the Forestry Research Institute of Nigeria, Ibadan, Nigeria. Grains of unmilled upland rice (*O. sativa*, Linn; ITA 150) were obtained from the International Institute for Tropical Agriculture, Ibadan, Nigeria.

### 2.2. Methods

#### 2.2.1. Preparation of natural and pregelatinized starches

Isolation and purification of the natural starches of white T. yam, yellow T. yam and rice were carried out using established procedures (Nkala et al., 1999; Perez-Sira, 1997; Young, 1984).

All the four starches were fully pregelatinized using the method described by some workers and in The Pharmaceutical Codex (Alebiowu & Itiola, 2002; Herman et al., 1989; The Pharmaceutical Codex, 1979). A quantity (100 g) of each of the natural starches was dispersed in 100 mL of distilled water and then heated at 55 °C with constant stirring for 10 min to form a paste which was crisp-dried in an oven at 60 °C for 48 h. The resultant mass was pulverized in a laboratory mill (Christy and Norris Ltd., UK). Each sample was passed through a number 120 mesh sieve (125 µm) and then stored in air-tight amber-coloured bottles.

#### 2.2.2. Determination of pH and moisture content

The pH of 20% slurry of each starch sample was determined on a pH meter (Kent Industrial Measurements, England). The moisture content was determined by drying 10 g of each sample in a tarred dish at 100 ± 2 °C to constant weight. The percentage loss in weight was recorded as moisture content.

#### 2.2.3. Amylose content determination

The procedure of Juliano (1971) was used to determine the amylose content (%) of each starch sample. A quantity of each sam-

ple (100 mg) was weighed into a 100 mL volumetric flask and heated for 10 min with 1 mL of 95% ethanol and 9 mL of 1 N caustic soda in a boiling water bath to gelatinize the starch. The material was then cooled and made up to 100 mL with distilled water. A portion of the starch solution (5 mL) was pipetted into a 100 mL volumetric flask. 1 mL of 1 N acetic acid and 2 mL of iodine solution were added and made up to volume (100 mL) with distilled water. After 20 min, the material was shaken and spectrophotometric absorbance was determined at 620 nm:

$$\begin{aligned} \% \text{Amylose} &= 3.06^* \times \text{Absorbance} \times 20 \\ *3.06 &\text{ is conversion factor} \end{aligned} \quad (3)$$

#### 2.2.4. Water absorption capacity, solubility and swelling power determination

The method used by Solsulski (1962) was employed to determine the water absorption capacity for each starch powder. Percent solubility and the swelling power were determined by the methods described by Leach, McCowen, and Schoch (1959) and Bowen and Vadino (1984), respectively.

#### 2.2.5. Viscoamylography

Viscosity profiles of the starch materials were obtained using a heating and cooling viscometer, series 3RVA (Rapid Visco Analyser) coupled with Thermocline for Windows software (Newport Scientific Pty. Ltd. Warriewood, NSW Australia). The test proceeded and terminated automatically. Heating of the slurry in the equipment was done under a constant rate of shear, and the increase in viscosity of material was measured as torque on the spindle and a curve was traced (Thomas & Atwell, 1999).

Various parameters were determined from the trace:

- Peak viscosity – maximum viscosity of material developed soon after the heating portion of the test;
- Peak time – time at which the peak viscosity occurred;
- Peak temperature – temperature at which peak viscosity occurred;
- Trough viscosity – lowest viscosity after the peak viscosity just before it begins to increase again;
- Breakdown – peak viscosity minus trough viscosity;
- Final viscosity – viscosity at the end of the test;
- Setback from trough – final viscosity minus trough viscosity (retrogradation);
- Setback from peak – final viscosity minus peak viscosity.

#### 2.2.6. Particle size and shape determination

The particle size distribution of each sample suspended in ethanol was determined using an optical microscope (Olympus Optical Co., Japan). Four hundred (400) particles were measured and the mean projected particle diameter ( $\bar{d}$ ) was determined for each starch material.

Particle shape was determined by photomicrography. A small quantity of the material was dispersed in ethanol. A drop of this dispersion was placed on a glass slide, covered with a slip and examined under 100× objective in bright field of an optical microscope (Olympus Optical Co., Japan). Photomicrographs of the particles were then taken on an ND 2a Leica camera fitted on the microscope.

The shape coefficient  $\alpha$ , for each starch sample was determined from the following expression (Nikolakakis & Pilpel, 1985):

$$\alpha = \rho_p \text{ Sw } d_e + \text{ELR} \quad (4)$$

Where  $d_e$  is the Heywood's equivalent diameter (m) and ELR is the elongation ratio,  $L/B$  – that is, the ratio of the values of average particle length,  $L$  (m) and breadth  $B$  (m):

$$D_e = \left[ \frac{0.77 \times 4 \times L \times B^{1/2}}{\pi} \right] \quad (5)$$

### 2.2.7. Measurement of densities

Particle density ( $\rho_p$ ) values for all the materials were determined by the pycnometer method with xylene as the displacement fluid. The loose bulk density ( $\rho_0$ ) was determined for each sample by pouring 30 g of the powder at an angle of 45° through a glass funnel into a 50 mL glass measuring cylinder of diameter of 28 mm (Itiola, 1991). The precompression density,  $R_0$ , of each starch powder was obtained from the ratio of loose bulk density to its particle density. The Hausner's ratio (Herman et al., 1989), determined as the ratio of the initial bulk volume to the tapped volume, was obtained by applying 100 taps to 30 g of each starch sample in a graduated cylinder at a standardized rate of 38 taps per minute (British Standard 1460, 1970). All determinations were done in quadruplicate.

### 2.2.8. Preparation of starch tablets

480 mg of each starch sample was compressed for 1 min with nine predetermined pressures between 28 and 226 MNm<sup>-2</sup>, on a Carver hydraulic hand press (Model C, Carver Inc. Wisconsin, USA), using a 10.5 mm die and flat-faced punches lubricated with a 2% w/v dispersion of magnesium stearate in ether–ethanol (1:1) prior to each compression. Tablets of 3.68 ± 0.22 mm thickness at zero porosity as calculated from particle density values were obtained. Tablets with a central hole (1.59 mm diameter) were made using an upper punch with a hole through the centre and a lower punch fitted with a pin (Itiola, 1991). The tablets were stored over silica gel for 48 h to allow for elastic recovery and hardening and to prevent falsely low yield values.

The compressibility of the starches was investigated by constructing Heckel and Kawakita plots for all the samples. Values of relative density ( $R$ ) of prepared tablets were calculated from the weights and dimensions determined to within ±1 mg and

0.01 mm, respectively, and the particle density of the solid material:

$$R = w/V_t \rho_p \quad (6)$$

where  $V_t$ , the volume (cm<sup>3</sup>) of the tablet, included the hole when present.

The tensile strengths of the normal tablet and apparent tensile strengths of those containing a central hole were obtained at room temperature by diametral compression using a Monsanto hardness tester and by applying Eq. (1) (Fell & Newton, 1970). The brittleness of the tablets was calculated using Eq. (2).

## 3. Results and discussion

Moisture typically equilibrates to about 12% or less in a starch powder (Thomas & Atwell, 1999). In the present work, the moisture content of the starches was lower than 10%.

The amylose content of the starches was less than 50% implying that the amylopectin content was more than 50% (Thomas & Atwell, 1999), but the amylose content of starches is important because amylose largely determines the gelling ability of starches. The natural starches had higher amylose content than the pregelatinized starches and would be expected to produce harder gels. It is notable that corn starch had the highest values of amylose content with the trifoliolate yams having the least values.

The particles of the starches were generally ovoid to spherical as shown in the photomicrograph presented in Fig. 1. Values for physico-chemical properties of the starches are given in Table 1. The pregelatinized starches had higher values of mean projected diameter ( $\bar{d}$ ) than the natural starches as has been observed for fully pregelatinized starches prepared by other investigators (Alebiowu & Itiola, 2002; Herman et al., 1989). Corn starch had considerably higher  $\bar{d}$  values than the experimental starches. The values of the particle shape coefficient ( $\alpha$ ) for the natural starches were higher than those for the pregelatinized starches in agreement with the observation of Alebiowu and Itiola (2002) for other starches. It is also notable that corn starch had higher  $\alpha$  values than the experimental starches.

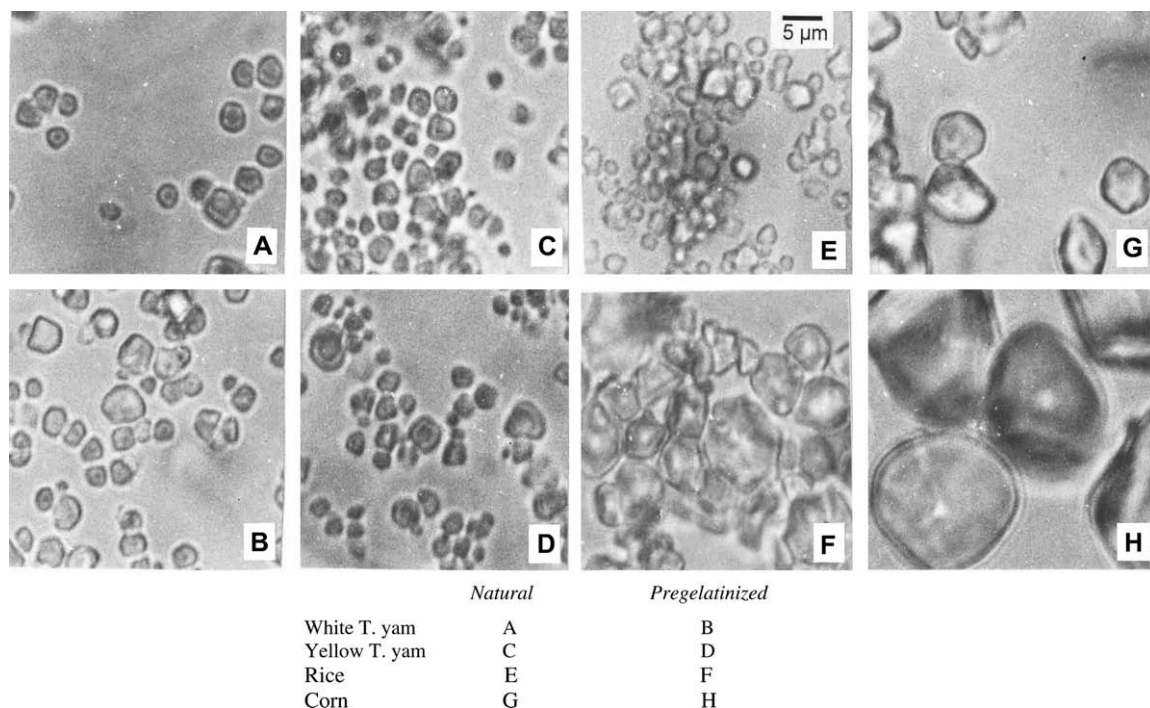


Fig. 1. Photomicrographs of the starches.

**Table 1**  
Physico-chemical properties of the starches.

Nature of starch	Form of starch	Mean projected diameter, $\bar{d}$ ( $\mu\text{m}$ )	Particle density, $\rho_p$	Moisture content (%)	Amylose content (%)	Average particle shape factor, $\alpha$	Hausner's ratio	Swelling power	Solubility (%)	Water absorption capacity (g/100 g)	pH
White T. yam	Natural	2.50	1.558	9.26	13.96	5.14	1.269	4.51	3.88	18.09	6.08
	Pregel	3.70	1.604	5.79	7.47	5.08	1.180	15.11	16.00	120.92	6.32
Yellow T. yam	Natural	2.80	1.547	8.54	13.71	5.26	1.370	5.28	4.85	27.14	5.96
	Pregel	3.80	1.596	5.77	7.96	5.24	1.208	11.16	12.00	112.39	6.15
Rice	Natural	3.30	1.421	9.29	15.12	5.45	1.406	5.28	4.91	18.29	6.02
	Pregel	5.90	1.541	5.86	10.53	5.32	1.301	10.93	7.00	94.77	6.40
Corn	Natural	13.80	1.479	9.30	24.05	6.77	1.364	5.78	4.88	32.08	5.96
	Pregel	18.50	1.520	6.02	10.77	6.09	1.199	18.03	19.00	102.63	6.05

The Hausner's ratio provides an indication of the degree of densification, which could result from vibration of the feed hopper, for example, during tableting, with higher values predicting significant densification of powders. The ranking for the Hausner's ratio of the starches was generally rice > yellow T. yam > corn > white T. yam. Furthermore, pregelatinized starches had lower values of the Hausner's ratio suggesting better flowability than natural starches. The values of particle density for the pregelatinized starches were however, higher than those for the natural starches.

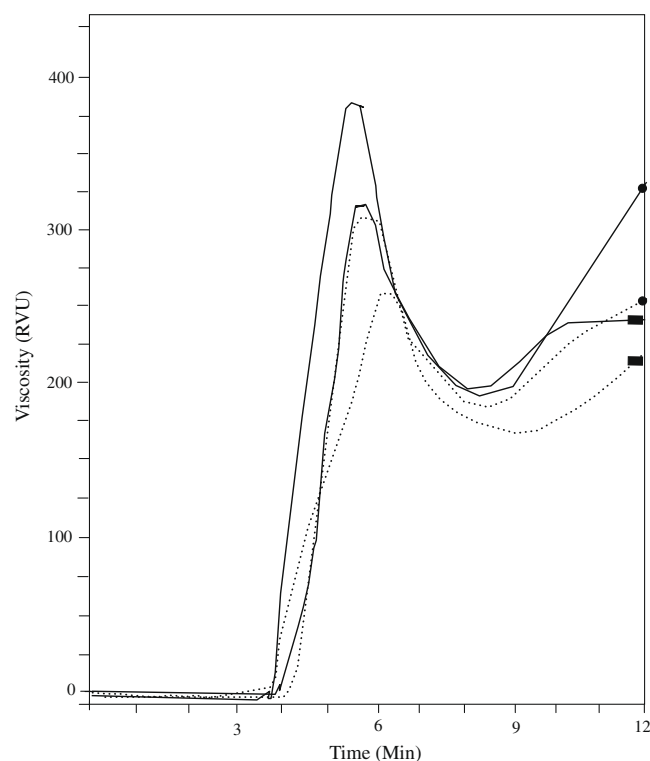
The pregelatinized starches showed higher swelling ability, percentage solubility and water absorption capacity than the natural starches. Thus, as expected, pregelatinization increased the cold-water swellability of the starches (Alebiowu & Itiola, 2002; Visavarungroj & Remon, 1991). This effect is probably due to disruption of the starch grains during pregelatinization, which would release amylopectin which is partially responsible for the swelling of starch (Thomas & Atwell, 1999). It is also notable that corn starch generally exhibited the highest swelling ability and percentage solubility. The pH values for the pregelatinized starches were also higher than those for the natural starches.

Fig. 2 shows representative viscosity profiles for the natural and pregelatinized starches of white and yellow T. yam starches. The viscosity profile is largely a reflection of the changes in the starch granules that occur during the heating and cooling cycle in the Rapid Visco Analyzer (Thomas & Atwell, 1999). During the initial heating phase, a rise in viscosity is recorded as granules begin to swell. A peak viscosity is obtained at which point there is a majority of fully swollen, intact granules. During the high temperature hold phase (95 °C), the granules begin to break down, polymer solubilization continues, and molecular alignment occurs within the shear field of the instrument. At this point, a drop in viscosity is recorded (Breakdown viscosity). During the subsequent cooling phase, solubilized amylose and amylopectin polymers begin to reassociate (retrogradation) and another rise in viscosity is traced to a final viscosity. This second rise in viscosity is usually referred to as set-back from peak and the difference between final and trough viscosities is called set-back from trough. Low values of these parameters imply that the materials are resistant to changes in viscosity during the heating and cooling cycle. In addition, the peak time and temperature of viscosity indicate an inverse relative sensitivity of the starches to heat. The time and temperature will vary depending on the nature and form of starch involved.

Viscosity parameters for all the starches are given in Table 2. It is seen that both natural and pregelatinized forms of corn starch had the lowest peak time and temperature. This indicates that the corn starches were the most sensitive to heat and would form a gel or paste at a lower temperature than the experimental starches. The ranking of peak temperature for both forms of the starches was corn < rice < white T. yam < yellow T. yam, showing that the yam starches were the least sensitive to heat. The ranking

of peak time was less clear-cut with rice starch having the highest peak time. Pregelatinized starches were less sensitive to heat as they had higher values of peak temperature and time than the natural starches. Three other important parameters in characterizing the viscosity profiles of the starches are the peak viscosity, breakdown viscosity and final viscosity. The importance of trough viscosity is essentially in its use to calculate the breakdown viscosity, which indicates the extent of granule breakdown and a consequent drop in viscosity during the hold phase of viscoamylography. Trough viscosity is also important in calculating the set-back from trough (retrogradation).

The peak and breakdown viscosity values are measures of the stability of the starch materials showing the resistance to breakdown of their granules. On the other hand, the final viscosity is a direct measure of the viscosity of the gel formed after retrogradation. The natural starches exhibited higher values of all the three parameters than the pregelatinized starches. Table 2 also shows that the natural starches exhibited higher set-back from trough (retrogradation) than pregelatinized starches except for rice starch.



**Fig. 2.** Viscoamylograph traces (viscosity profiles) for the natural (—) and pregelatinized (---) starches of white (●) and yellow (■) T. yams.



**Table 2**

Parameters obtained from viscoamylography.

Nature of starch	Form of starch	Peak viscosity (RVU)	Peak time (min)	Trough viscosity (RVU)	Breakdown (RVU)	Peak temperature (°C)	Final viscosity (RVU)	Setback from trough (RVU)	Setback from peak (RVU)
White T. yam	Natural	376.20	5.44	216.08	160.12	80.60	295.67	79.59	−80.58
	Pregelatinized	304.18	5.87	189.83	114.35	81.80	236.75	46.92	−67.83
Yellow T. yam	Natural	309.25	5.21	208.83	100.42	84.10	452.00	243.17	142.75
	Pregelatinized	260.50	6.03	181.33	79.17	85.60	417.25	235.92	156.75
Rice	Natural	306.08	6.91	211.75	94.33	76.95	244.92	33.17	−16.16
	Pregelatinized	235.67	6.97	161.75	73.92	79.85	219.58	57.83	−16.09
Corn	Natural	356.75	5.05	236.50	120.25	74.65	299.75	63.25	−57.00
	Pregelatinized	282.75	5.18	202.83	79.92	76.70	256.17	53.34	−26.58

The results show that the natural starches are more susceptible to changes in viscosity during the heating and cooling cycle. The natural starches suffered more retrogradation than the pregelatinized starches as shown by their higher breakdown viscosity. This is due to the higher content of amylose in the natural starches (Freitas et al., 2004). Amylose consists of linear molecules, which have a greater tendency to reassociate and form hydrogen bonds than the larger non-linear amylopectin molecules (Thomas & Atwell, 1999).

For the different starches, rice starch had the lowest values of peak, breakdown and final viscosities suggesting that its granules are the least susceptible to breakdown. On the other hand, while white T. yam had the highest values of peak and breakdown viscosities, yellow T. yam starch had the highest values of final viscosity. It is also notable that yellow T. yam starch had the highest values of set-back from trough (retrogradation) and set-back from peak. The values of these two parameters for yellow T. yam starch were much higher than their values for the other starches, which were more comparable. In fact, all the values of set-back from peak for the other three starches – rice, corn and white T. yam – were negative, with only yellow T. yam starch showing a positive value indicating that its final viscosity was higher than its peak viscosity.

Fig. 3 shows Heckel plots for the starches. Values of mean yield pressure,  $P_y$ , were calculated from the region of the plots showing the highest correlation coefficient for linearity of  $>0.996$  for all the starches (generally between 84.90 and 226.40 MNm<sup>−2</sup>). The intercept,  $A$ , was determined from the extrapolation of the region used for the calculation of  $P_y$ . The values of  $R_a$  and  $R_b$  were also calculated. Values of  $P_y$ ,  $R_0$ ,  $R_a$  and  $R_b$  for all the starches are presented in Table 3.

The values of  $R_0$ ,  $R_a$  and  $R_b$  for the pregelatinized starches were higher than those for the natural starches indicating that the pregelatinized starches exhibited higher total degree of packing both during die filling (zero pressure) and at low pressures. The ranking for the total degree of packing ( $R_a$ ) for all the starches was generally yellow T. yam > rice > white T. yam > corn.

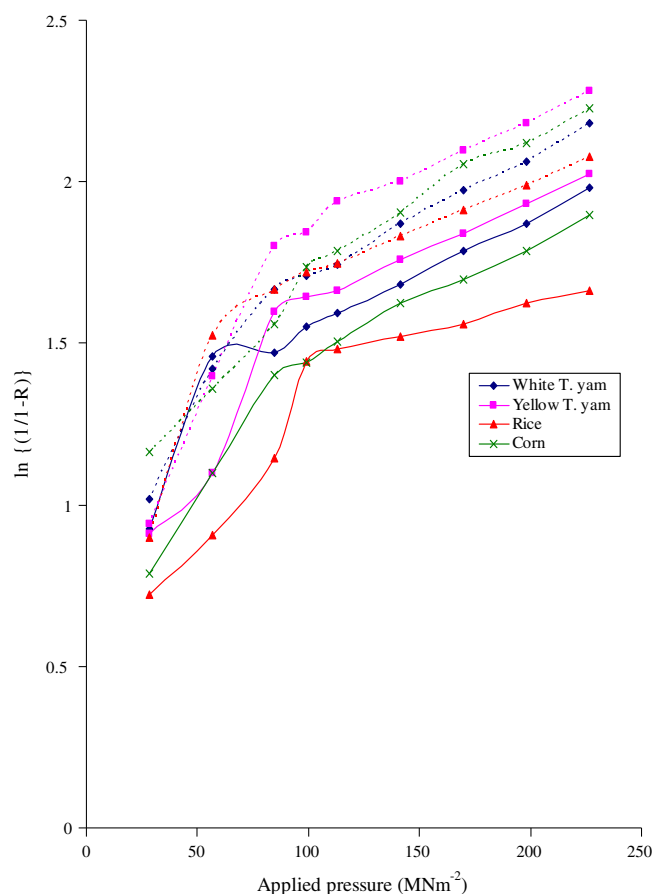
The mean yield pressure,  $P_y$ , is inversely related to the ability of a material to deform plastically under pressure. The ranking of  $P_y$  for the starches was generally corn < white T. yam < yellow T. yam < rice. Furthermore, the values of  $P_y$  for the pregelatinized starches were lower than those for the natural starches, implying that the onset of plastic deformation in the pregelatinized starches occurred at lower pressures. The lowest yield pressures ( $P_y$ ) for the corn starches which imply the fastest rate of plastic deformation may also explain why the corn starches were the most heat sensitive.

Fig. 4 shows Kawakita plots for the starches. 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 of the plots, respectively. Values of  $1 - a$  give the initial relative density of

the starches  $R_i$ , while  $P_k$  values were obtained from the reciprocal of values of  $b$ .

The value of  $R_i$  and  $P_k$  are included in Table 4. The values of  $R_i$  which is a measure of the packed initially density of the starches (Odeku & Itiola, 1998), are seen to be higher for the pregelatinized starches than for the natural starches. These values are also seen to be generally higher than the corresponding values of the loose initial relative density,  $R_0$ . This is in agreement with previous findings (Alebiowu & Itiola, 2002; Odeku & Itiola, 1998). The ranking for the values of  $R_i$  was generally rice > yellow T. yam  $\approx$  corn > white T. yam.

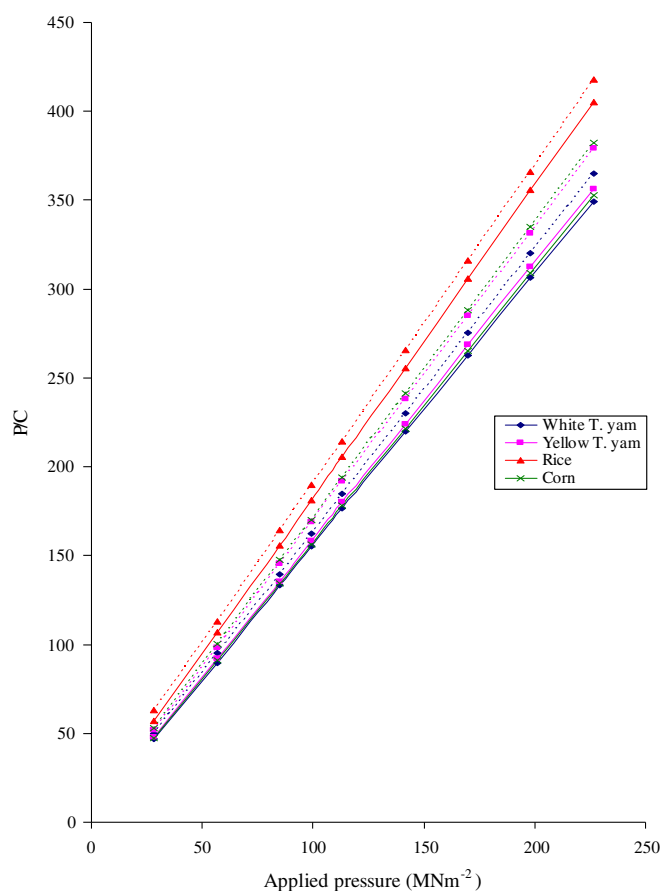
The values of  $P_k$ , which is an inverse measure of the total amount of plastic deformation occurring during the compression process (Odeku & Itiola, 1998), were found to be lower for the natural starches than the pregelatinized starches. It has been established that generally the lower the  $P_k$  value, the more the total

**Fig. 3.** Heckel plots for natural (—) and pregelatinized (---) starches.

**Table 3**

Parameter obtained from density measurements and from Heckel and Kawakita plots for the starches.

Nature of starch	Form of starch	Heckel plots				Kawakita plots	
		$R_0$	$P_y$	$R_a$	$R_b$	$R_d(1-a)$	$P_k$
White T. yam	Natural	0.300	288.8	0.697	0.397	0.346	2.264
	Pregelatinized	0.337	272.6	0.740	0.403	0.372	3.019
Yellow T. yam	Natural	0.317	335.8	0.738	0.421	0.359	2.346
	Pregelatinized	0.318	301.5	0.784	0.466	0.399	3.289
Rice	Natural	0.259	592.4	0.722	0.463	0.431	3.939
	Pregelatinized	0.273	349.2	0.760	0.487	0.441	6.820
Corn	Natural	0.355	289.1	0.671	0.316	0.351	2.347
	Pregelatinized	0.382	255.3	0.741	0.359	0.399	3.480

**Fig. 4.** Kawakita plots for natural (—) and pregelatinized (---) starches.**Table 4**

Values of tensile strength and brittle fracture index for the starches at relative density of 0.90.

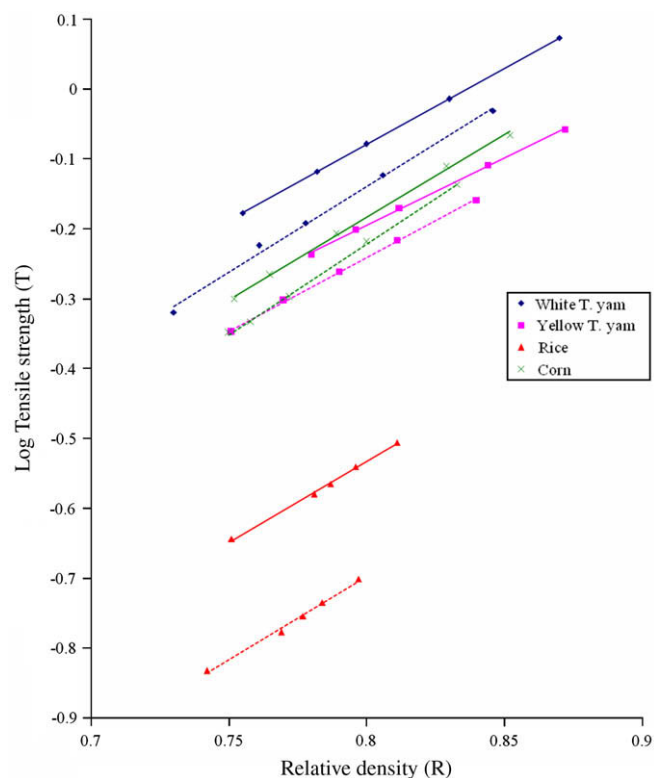
Nature of starch	Form of starch	Tensile strength, $T$ (MNm <sup>-2</sup> )	Brittle fracture index (BFI)
White T. yam	Natural	1.375	0.039
	Pregelatinized	0.851	0.022
Yellow T. yam	Natural	0.995	0.033
	Pregelatinized	0.739	0.026
Rice	Natural	0.498	0.213
	Pregelatinized	0.446	0.170
Corn	Natural	1.128	0.017
	Pregelatinized	0.634	0.012

plastic deformation occurring during compression (Alebiowu & Itiola, 2002; Odeku & Itiola, 1998). The ranking of  $P_k$  for the starches was white T. yam < yellow T. yam < corn < rice.

The results of the tensile tests on the starches were found to fit the general equation:

$$\log T(\text{or } T_h) = mR + c \quad (7)$$

with a correlation coefficient of >0.990.  $m$  and  $c$  were constants which depended on the nature and form of starch involved and on whether or not the tablet had a hole. Fig. 5 shows representative plots of log tensile strength versus relative density for the natural and pregelatinized forms of white and yellow T. yam starches. The tensile strength of starch tablet with a hole was lower than that of the same without a hole, as the hole acts as a stress concentrator. It can also be seen from the Figure that tablets made from the pregelatinized form of the starch had lower  $T$  values than tablets made from natural starches at the same relative density. This is in agreement with previous findings (Alebiowu & Itiola, 2002).

**Fig. 5.** Log tensile strength against relative density for natural starch tablets with (---) and without (—) a central hole.

Values of  $T$  and BFI for the starches at relative density of 0.90 which is representative of commercial tablets are presented in Table 4. The ranking of  $T$  for the starches was generally white T. yam > corn > yellow T. yam > rice. Also, the rankings of the BFI for the natural and pregelatinized starches were corn < yellow T. yam < white T. yam < rice; and corn < white T. yam < yellow T. yam < rice, respectively. The pregelatinized starches had lower BFI values than the natural starches and consequently possessed a higher ability to reduce the lamination tendency in tablets than natural starches.

It is of interest to see whether the rheological properties of the starches show any relation to the tableting properties of the starches. It is notable that the natural starches with higher amylose content, and consequently with the ability to form harder gels than the pregelatinized starches (Thomas & Atwell, 1999), gave tablets with higher tensile strength. It may also be of relevance that the natural starches exhibited higher viscosity values in the heating cycle. The pregelatinized starches with more ability to resist breakdown in the viscoamylography also gave tablets that are less brittle and exhibited lower ability to cap or laminate than tablets made from natural starches. However, when the individual starches are considered, the ranking of the various material properties do not correlate with the ranking of the tablet properties. It would appear therefore that rheological properties of the starches are probably of limited value to predict properties of tablets produced from the starches.

#### 4. Conclusion

The results of the present work provide some insight into the material and compaction properties of the experimental starches. The fact that the experimental starches showed similar properties to corn starch for some of the parameters evaluated suggests that the experimental starches may serve as suitable alternatives to corn starch for particular purposes.

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#### References

- Alebiowu, G., & Itiola, O. A. (2001). Effects of natural and pregelatinized sorghum, plantain and corn starch binders on the compressional characteristics of a paracetamol tablet formulation. *Pharmaceutical Technology*, 26–30 [Suppl. Ed: Drug Delivery].

- Alebiowu, G., & Itiola, O. A. (2002). Compressional characteristics of native and pregelatinized forms of sorghum, plantain and corn starches and the mechanical properties of their tablets. *Drug Development & Industrial Pharmacy*, 28(6), 663–672.
- Atichokudomchai, N., & Varavinit, S. (2003). Characterization and utilization of acid-modified cross-linked Tapioca starch in pharmaceutical tablets. *Carbohydrate Polymers*, 53, 263–270.
- Ayernor, G. S. (1985). The yam (*Dioscorea*) starches. In Godson Osuji (Ed.), *Advances in yam research – The biochemistry and technology of the yam tuber* (pp. 79–88). Pub. Biochemical Society of Nigeria in Collaboration with Anambra State University of Technology.
- Bowen, F. E., & Vadino, W. A. (1984). A simple method for differentiating starches. *Drug Development & Industrial Pharmacy*, 10, 505–511.
- British Standard 1460 (1970). British Standard Institution, London.
- Fell, J. T., & Newton, J. M. (1970). Determination of tablet strength by diametral compression test. *International Journal of Pharmaceutics*, 59, 688–691.
- Freitas, R. A., Paula, R. C., Feitosa, J. P. A., Rocha, S., & Seirakowski, M. R. (2004). Amylose contents, rheological properties and gelatinization kinetics of yam (*Dioscorea alata*) and cassava (*Manihot utilissima*) starches. *Carbohydrate Polymers*, 55, 3–8.
- Heckel, R. W. (1961). Density–pressure relationships in powder compaction. *Transaction of the Metallurgical Society of AIME*, 221, 671–675.
- Heistand, E. N., Wells, J. E., Poet, C. B., & Ochs, J. F. (1977). Physical processes of tableting. *Journal of Pharmaceutical Sciences*, 66, 510–519.
- Herman, J., Remon, J. P., & De-Vilder, J. (1989). Modified starches as hydrophilic matrices for controlled oral delivery I. Production and characterization of thermally modified starches. *International Journal of Pharmaceutics*, 56, 51–63.
- Itiola, O. A. (1991). Compressional characteristics of three starches and the mechanical properties of their tablets. *Pharmacy World Journal*, 8, 91–94.
- Juliano, B. O. (1971). A simplified assay for milled-rice amylose. *Cereal Science Today*, 16, 334–340.
- Kawakita, K., & Ludde, K. H. (1970/71). Some considerations on powder compression equations. *Powder Technology*, 4, 61–68.
- Leach, H. W., McCowen, L. D., & Schoch, T. J. (1959). Structures of the granules. I swelling and solubility patterns of various starches. *Cereal Chemistry*, 36, 534–542.
- Lin, C., & Cham, T. (1995). Compression behaviour and tensile strength of heat-treated polyethylene glycols. *International Journal of Pharmaceutics*, 118, 169–179.
- Maize starch pregelatinized. In *The pharmaceutical codex* (11th ed., p. 510). London: The Pharmaceutical Press, 1979.
- Nikolakakis, L., & Pilpel, N. (1985). Effect of particle shape on the tensile strength of powders. *Powder Technology*, 42, 279–283.
- Nkala, D., Sibanda, S., Tomasik, P., & Palasinski, M. (1999). Isolation and properties of starch from wild yam from Zimbabwe. *Starch/Stärke*, 46(3), 85–88.
- Odeku, O. A., & Itiola, O. A. (1998). Evaluation of Khaya gum as a binder in a paracetamol tablet formulation. *Pharmacy & Pharmacology Communications*, 4, 183–188.
- Perez-Sira, E. (1997). Characterization of starch isolated from plantain (*Musa paradisiacal normalis*). *Starch/Stärke*, 49(2), 45–49.
- Solsulski, F. W. (1962). The centrifuge method for determining flour absorptivity in hard red spring wheats. *Cereal Chemistry*, 39, 344–350.
- Thomas, D. J., & Atwell, W. A. (1999). In *Starches: Practical guides for the food industry* (pp. 19–22). St. Paul, Minnesota, USA: American Association of Cereal Chemists.
- Visavarungroj, N., & Remon, J. P. (1991). An evaluation of hydroxypropyl starch and disintegrant and binder in tablet formulation. *Drug Development & Industrial Pharmacy*, 17, 1389–1396.
- Watt, J. M., & Breyer-Brandwijk, M. G. (1962). *The medicinal and poisonous plants of southern and eastern Africa*. London: E&A Livingstone. p. 385.
- Young, A. H. (1984). Fractionation of Starch. In R. L. Whistler, J. N. BeMiller, & E. F. Paschal (Eds.), *Starch chemistry and technology* (2nd ed., pp. 249–283). London: Academic Press.