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RESEARCH PAPER

Compressional Characteristics of Native and Pregelatinized Forms of Sorghum, Plantain, and Corn Starches and the Mechanical Properties of Their Tablets

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ABSTRACT

A study was made of the compressional characteristics of native and pregelatinized forms of sorghum, plantain, and corn starches and the mechanical properties of their tablets. Compressional characteristics were analyzed using density measurements and the Heckel and Kawakita plots. Pregelatinized starches exhibited more densification than native starches during die filling and at low pressures. The ranking for the mean yield pressure (P_y) values for the starches was plantain < corn < sorghum, with the pregelatinized starches having lower values than the native starches. The ranking for the values of another pressure term, P_k—an inverse measure of plasticity, was corn < plantain < sorghum, but with the native starches having the lower values. For the tablets, the ranking for values of tensile strength (T) was corn > plantain > sorghum, while the ranking for the brittle fracture index (BFI) was plantain > corn > sorghum. Tablets made from pregelatinized starches had lower T and BFI values than those made from native starches. The results suggest that pregelatinization of the starches facilitated faster onset of plastic deformation but reduced the amount of plastic deformation which occurred during the compression process.

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INTRODUCTION

Starch is a multifunctional excipient in tablet formulation which can be used as binder, disintegrant, or filler, due to its suitable physicochemical properties and relative inertness. In recent years, pharmaceutical scientists have been giving increasing attention to the extraction, development, and use of starches in the formulation of dosage forms (1). Native starches have also been pregelatinized in order to produce cold water-swellable starches and to increase the flowability of the starches (2). Pregelatinization can be carried out using thermal methods (3), and such modification would be expected to affect various properties of starches, including their compressibility.

Sorghum and plantain starches obtained from *Sorghum bicolor* (Poaceae) and *Musa paradisiaca* (Musaceae), respectively, have been investigated as binders and disintegrants in tablet formulations (1,4). However, it appears that no attempt has been made to study the compressional properties of these starches with a view to providing more information on their potential usefulness in tablet formulation and production.

In the present work, a study has been made of the compressional properties of native and pregelatinized sorghum and plantain starches and the mechanical properties of the resulting tablets. The results were compared with those obtained using official corn starch BP. Compressional characteristics were analyzed using the Heckel and Kawakita equations (5,6), while the mechanical properties of the tablets were determined using their tensile strength and brittle fracture index (BFI) (7,8).

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:

$$\ln[1/(1-D)] = KP + A \tag{1}$$

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 the intercept A, the relative density, D_a, can be calculated using the following equation (9):

$$D_a = 1 - e^{-A} \tag{2}$$

The relative density of the powder at the point when the applied pressure equals zero, D_0, 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_0:

$$D_b = D_a - D_0 \tag{3}$$

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

$$C = (V_0 - V_p)/V_0 = abP/(1 + bP) \tag{4}$$

The equation, in practice, can be rearranged to give:

$$P/C = P/a + 1/ab \tag{5}$$

where V_0 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 provides an inverse measure of the deformability of the particles during compression (10,11) and has been shown to be the pressure required to reduce the powder bed by 50% (12,13).

Tensile strength and brittle fracture index are two important parameters which have been used as measures of the bond strength and lamination tendency of tablets respectively (14–16). The BFI is defined as (8):

$$\text{BFI} = 0.5[(T/T_0) - 1] \tag{6}$$

where T is the tensile strength of the tablet without a hole and T_0 is the apparent tensile strength of the tablet when a hole is present at its center—both at the same relative density. The hole acts as a built-in stress concentrator defect. The BFI is a measure of localized stress relief within the tablet (at the edge of the hole) by plastic deformation. A low value of the BFI indicates the ability of the material to relieve localized stresses, while a value approaching unity indicates the tendency of the material to laminate or cap.

MATERIALS AND METHODS

Materials

The materials used were corn starch BP (BDH Chemicals Ltd., Poole, UK) and sorghum (*S. bicolor* L.) and plantain (*M. paradisiaca* L.) starches prepared in our laboratory. The pregelatinized

forms of the three starches were also prepared in our laboratory.

Preparation of Native and Pregelatinized Starches

Native sorghum and plantain starches were extracted from sorghum grains and unripe plantain fruits, respectively, according to established procedures (17). The fully pregelatinized forms of the two starches and of official corn starch BP were prepared as described in *The Pharmaceutical Codex* and by Herman et al. (3,18). An aqueous slurry of each starch was made with 100 g of starch powder in 100 mL deionized water and then heated at 55°C with stirring for 10 min. The resultant paste was crisp-dried in a hot air oven (Gallenkamp, Model OV-335, Vindon Scientific Ltd., Oldham, UK) at 60°C for 48 hr. The dried mass was powdered in a laboratory mill (Christy and Norris Ltd., Chelmsford, UK). All the starches were passed through a number 120 mesh sieve (125 μm) before use.

Determination of Starch Particle Size and Shape

Each of the starches was dispersed in Smith's starch reagent, in which the particles do not swell, before analysis. The particle size distribution of each starch was determined by optical microscopy (Leitz, Laborlux II, Germany) on 300 particles and was used to determine the mean projected particle diameter ($\vec{d}$).

Particle shape was determined by means of photomicrographs taken using a Leitz Dialux research microscope fitted with a Leica camera. The shape coefficient, α, of the particles of each starch was calculated from the expression (19):

$$\alpha = S_w \rho_s d_e + N \tag{7}$$

where S_w is the specific surface area of the particles ($m^2\,g^{-1}$) which was determined from the size distribution of the particles (20); d_e is the Heywood's equivalent diameter (μm) and is expressed as:

$$d_e = \left[\frac{0.77 \times 4 \times L \times B}{\pi}\right]^{1/2} \tag{8}$$

Here N, the elongation ratio, is L/B, where L and B are the arithmetic mean values of the particle length and breadth, respectively; and ρ_s is the particle density ($g\,cm^{-3}$) of the solid material.

Determination of Starch Densities

The particle density of each starch was determined using the pycnometer method with benzene as the displacement fluid (21). 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 21 mm and a volume of 50 mL (22,23). Determinations were done in triplicate. The relative density, D_0, of each starch powder was obtained from the ratio of its loose density to its particle density. The Hausner's ratio (3), 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 (24).

Determination of Starch Moisture Content

The moisture content of 10 g of each starch was determined on a wet-weight basis using an Ohaus infra-red moisture analyzer (Ohaus Scale Corporation, New Jersey).

Determination of Starch Swelling Capacity

The method described by Bowen and Vadino (25) was used. Five grams of each starch was poured into a 100-mL measuring cylinder and the bulk volume measured (V_1). Deionized water (90 mL) was added and the dispersion was well shaken for 5 min. Water was added to make 100 mL. The dispersion was allowed to stand for 24 hr before the sedimentation volume was read (V_2). The swelling capacity was calculated as V_2/V_1. Determinations were done in triplicate.

Determination of Starch Water Retention Capacity

This was determined using the method of Ring (26). To 5 g of each starch in a 100-mL measuring cylinder was added 90 mL of deionized water and the dispersion was well shaken for 5 min. Water was then added to make 100 mL. Fifteen milliliters of the dispersion was centrifuged (Optima Centrifuge type, BHG 500, Germany) for 25 min at 5000 rpm. The supernatant was discarded and the residue weighed (W_1). The residue was then dried at 70°C to constant weight (W_2) in a hot air oven. The water

retention capacity was computed as W_1/W_2. Determinations were done in triplicate.

Preparation of Tablets

Quantities (550 mg) of each starch powder, giving a tablet thickness of 3.04±0.11 mm at zero porosity as calculated from particle density values, were each compacted for 1 min with 10 predetermined loads ranging approximately from 20 to 160 MN m^{-2}, on a Carver hydraulic hand press (Model C, Carver Inc., Menomonee Falls, WI), using a 12.5-mm die and flat-faced punches lubricated with a 2% w/v dispersion of magnesium stearate in benzene before each compression. Tablets with a hole (1.54 mm diameter) at their center were made using an upper punch with a hole through the center and a lower punch fitted with a pin (14,23). After ejection, the tablets were stored over silica gel for 24 hr before tablet properties were determined, to allow for elastic recovery and hardening, and prevent falsely low yield values.

Determination of Tablet Properties

The weights (w) and dimensions of the tablets were determined to within ±1 mg and 0.01 mm, respectively, and their relative densities (D) were calculated using the equation:

$$D = w/V_t \times \rho_s \quad (9)$$

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

The tensile strengths of the normal tablets and apparent tensile strengths of those containing a hole were determined at room temperature by diametral compression (7) using a Monsanto crushing strength tester and by applying the equation:

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

where T (or T_0) is the tensile strength of the tablet (MN m^{-2}), F is the load (MN) needed to cause fracture, d is the tablet diameter (m), and t is the tablet thickness (m). Results were taken only from tablets which split cleanly into two halves without any sign of lamination. All measurements were made in triplicate.

The BFI values of the tablets were calculated using Eq. (6). Heckel plots of $\ln(1/1-D)$ vs. applied pressure (P), and Kawakita plots of P/C vs. P, were constructed for all the starches.

RESULTS AND DISCUSSION

Figure 1 shows photomicrographs of the starches. The particles of the starches were generally oval to spherical with the pregelatinized starches having bigger particles than the native starches. Table 1 gives values for various properties of the starches. The pregelatinized starches had higher values of mean projected particle diameter ($\bar{d}$), as has been observed for fully pregelatinized starches prepared by other investigators (3,27). The values of the particle shape coefficient (α) for the native starches were higher than those for the pregelatinized starches.

The Hausner's ratio provides an indication of the degree of densification which could result from vibration of the feed hopper, for example, during tabletting, with higher values predicting significant densification of powders. The ranking for the Hausner's ratio of the starches was generally corn > plantain > sorghum. Furthermore, the pregelatinized starches had lower values of the Hausner's ratio, suggesting better flowability than the native starches. The pregelatinized starches also had lower values of moisture content than the native ones. This agrees with the observation of Sanchez et al. (27) concerning the production of pregelatinized wheat starch from the native form. The values of particle density for the pregelatinized starches were, however, higher than those for the native starches.

The pregelatinized starches showed higher swelling ability and water retention capacity than the native starches. Thus, as expected, pregelatinization increased the cold water-swellability of the starches (2). This effect is probably due to distruption of the starch grains during pregelatinization, which would release amylopectin which is partially responsible for the swelling of starch (17).

Figure 2 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 >0.994 for all the starches (generally between 60 and 160 MN m^{-2}). The intercept, A, was determined from the extrapolation of the region used for the calculation of P_y. The values of D_a and D_b were calculated from Eqs. (2) and (3), respectively. Values of P_y, D_0, D_a, and D_b for all the starches are presented in Table 2.

The ranking of the values of D_0 for the starches was sorghum > corn > plantain. Furthermore, values of D_0 for the pregelatinized starches were

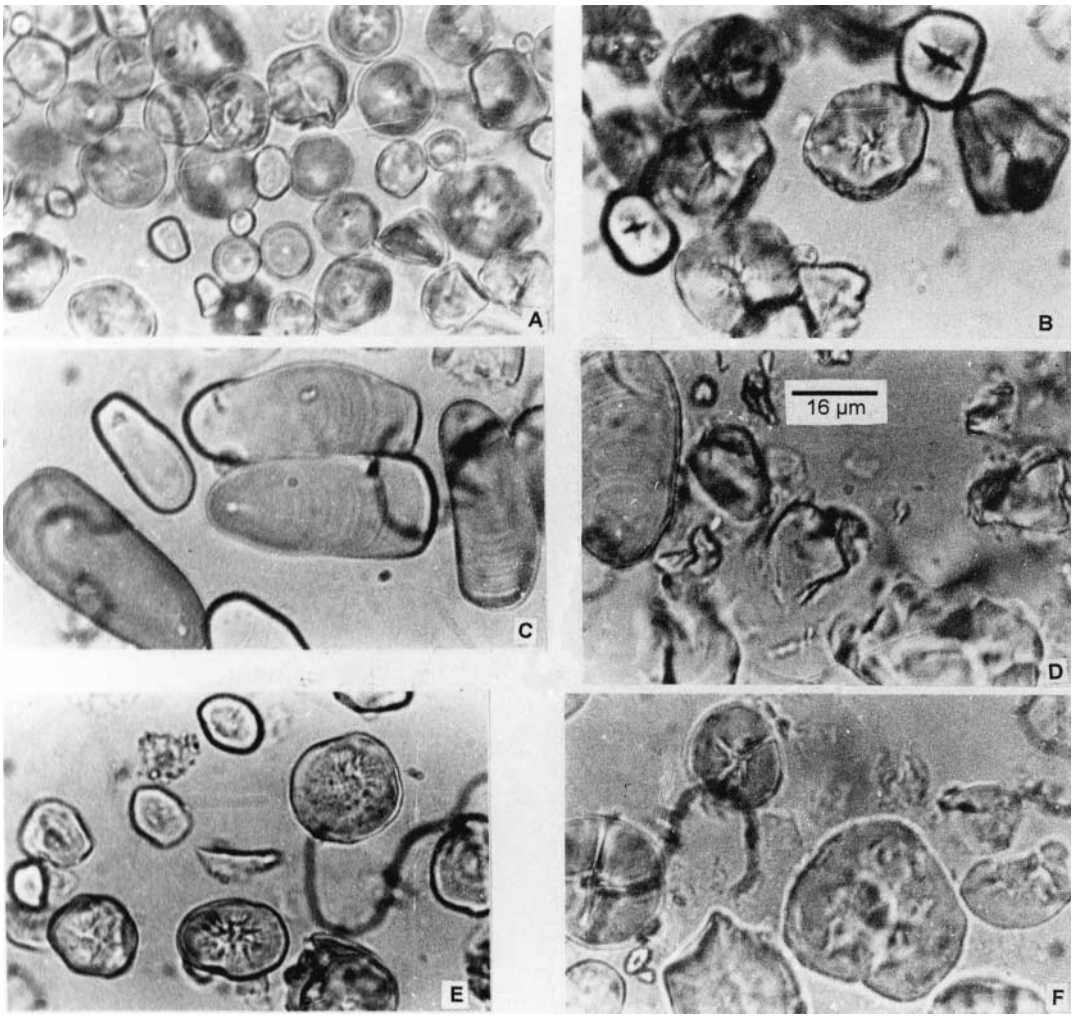

Figure 1. Photomicrographs of the starches: (A), native sorghum starch; (B), pregelatinized sorghum starch; (C), native plantain starch; (D), pregelatinized plantain starch; (E), native corn starch; (F), pregelatinized corn starch.

higher than those for the native starches, which implies that the pregelatinized starches exhibited a higher degree of packing in the die as a result of die filling.

The densification of the starches at low pressures is represented by D_b. The ranking of the values of D_b for the starches was plantain > corn > sorghum, the reverse of the ranking for D_0. The pregelatinized starches exhibited higher values of D_b than the native starches.

The values of D_a represent the total degree of packing achieved at zero and at low pressures [as implicit in Eq. (3)]. The ranking of D_a for the starches was plantain > corn > sorghum, with the pregelatinized starches exhibiting higher values than the native ones.

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 plantain < corn < sorghum. Furthermore, the values of P_y for the pregelatinized starches were lower than those for the native starches, implying that the onset of plastic deformation in the pregelatinized starches occurred at lower pressures.

Figure 3 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

Table 1

Microscopic and Physicochemical Characteristics of the Starches

Nature of Starch	Form of Starch	Mean Particle Diameter, $\bar{d}$ (μm)	Particle Density, ρ_s (g cm^{-3})	Heywood Equivalent Diameter, d_e (μm)	Elongation Ratio, N	Specific Surface Area, S_w (m^2 g^{-1})	Average Particle Shape Coefficient, α	Hausner's Ratio	Moisture Content (%)	Swelling Capacity	Water Retention Capacity
Sorghum	Native	16.7	1.493	14.29	1.34	0.0343	2.07	1.259	7.1	1.48	3.28
	Pregelatinized	17.9	1.531	15.85	1.19	0.0262	1.83	1.179	6.5	5.45	14.49
Plantain	Native	23.2	1.424	22.61	1.57	0.0367	2.75	1.302	7.8	1.53	3.43
	Pregelatinized	24.9	1.515	23.01	1.23	0.0352	2.45	1.174	6.9	5.27	10.36
Corn	Native	15.2	1.471	14.98	1.16	0.0434	2.17	1.348	6.8	1.57	3.03
	Pregelatinized	18.1	1.521	16.84	1.13	0.0392	2.13	1.216	6.1	5.78	9.42

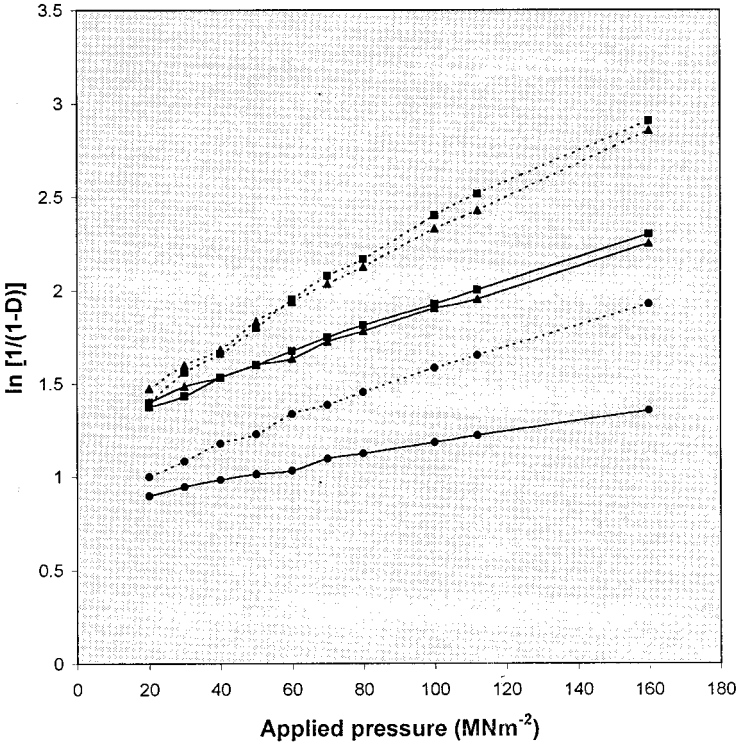

Figure 2. Heckel plots for native (——) and pregelatinized (- - - -) starches. ●, sorghum; ■, plantain starch; ▲, corn.

Table 2

Parameters Obtained from Density Measurements and from Heckel and Kawakita Plots for the Starches

Nature of Starch	Form of Starch	Heckel Plots				Kawakita Plots	
		D_0	P_y	D_a	D_b	D_i $(1-a)$	P_k
Sorghum	Native	0.345	323.6	0.580	0.235	0.427	4.536
	Pregelatinized	0.379	165.4	0.622	0.243	0.430	6.470
Plantain	Native	0.298	159.1	0.730	0.432	0.366	2.346
	Pregelatinized	0.317	104.7	0.771	0.454	0.398	3.095
Corn	Native	0.338	167.2	0.725	0.387	0.360	2.234
	Pregelatinized	0.353	109.4	0.752	0.399	0.414	2.980

were obtained from the slope and intercept of the plots, 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 included in Table 2. The values of D_i for the starches are seen to be higher than the corresponding values of D_0. Thus, the results are in agreement with the findings of Odeku and Itiola (16) that D_0 provides a measure of the loose initial relative density, while D_i provides a measure of the packed initial relative density of the material. The pregelatinized starches had higher values of D_i than the native starches. The ranking of D_i for the native starches was sorghum > plantain > corn, while the ranking for the pregelatinized starches was sorghum > corn > plantain.

Low values of P_k indicate materials that are soft and that readily deform plastically under

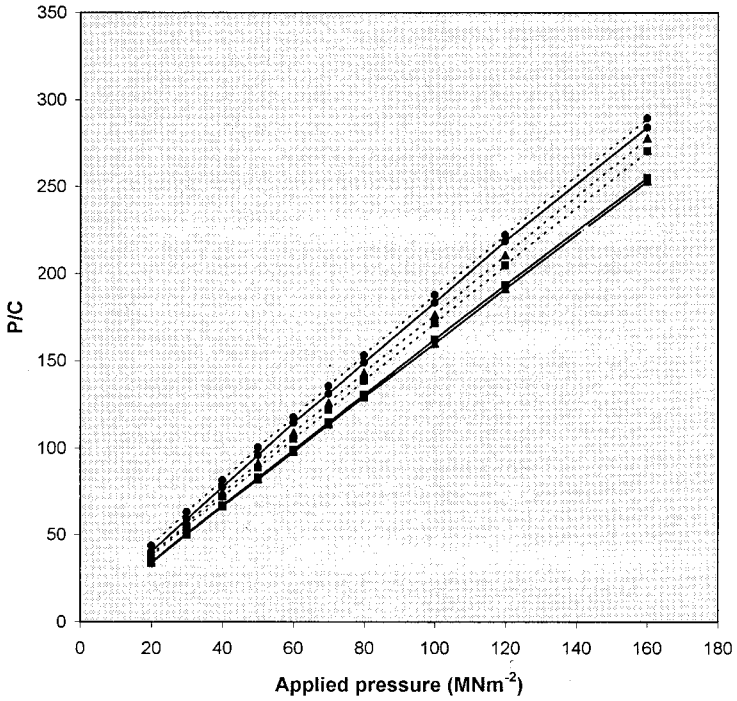

Figure 3. Kawakita plots for native (——) and pregelatinized (- - - -) starches. ●, sorghum; ■, plantain starch; ▲, corn.

Table 3

Tensile Strength and Brittle Fracture Index Values for the Starches at Relative Density of 0.90

Nature of Starch	Form of Starch	Tensile Strength ($MN\,m^{-2}$)	Brittle Fracture Index
Sorghum	Native	0.764	0.071
	Pregelatinized	0.721	0.054
Plantain	Native	1.026	0.139
	Pregelatinized	0.835	0.109
Corn	Native	1.077	0.099
	Pregelatinized	0.876	0.078

pressure. From Table 2, it is seen that the ranking of P_k for the starches was corn < plantain < sorghum. Furthermore, values of P_k for the native starches were lower than those for the pregelatinized starches.

Figure 4 shows representative plots of log tensile strength against relative density for plantain starch tablets. It can be seen that at all relative densities the tensile strength of a tablet with a hole was less than that of the same tablet without a hole, the hole acting as a stress concentrator (8). As expected (28), the results were found to fit the general equation:

$$\log T \text{ (or } T_0) = AD + B \tag{11}$$

with a correlation coefficient > 0.991. Here A and B are constants which depend on the nature and form of starch involved and on whether the tablet has a hole. It can also be seen from Fig. 4 that tablets made from the pregelatinized form of the starch have lower T values than tablets made from the native form at

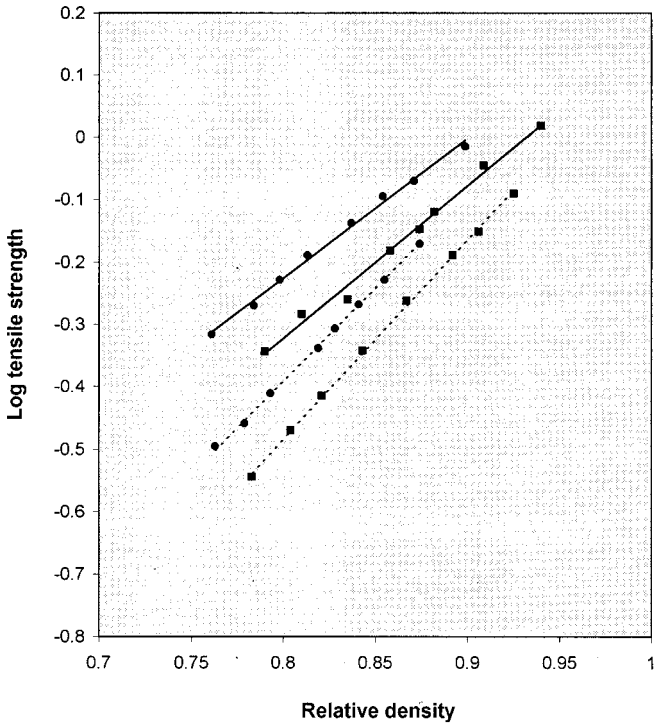

Figure 4. Log tensile strength vs. relative density for tablets made from plantain starch with hole (- - - -) and without a hole (——) at their center. ●, native; ■, pregelatinized.

the same relative density. This was generally the case for all the starches at relative density values >0.75. Lower crushing strength values have been observed for the pregelatinized forms of other starches by Bos et al. (29).

Values of T and BFI for the starches at $D=0.90$, which is representative of commercial tablets, are presented in Table 3. The ranking of T for the starches was corn > plantain > sorghum. For the BFI, the ranking was plantain > corn > sorghum. The pregelatinized starches had lower BFI values than the native starches, with the implication that the pregelatinized starches possessed a higher ability to reduce the lamination tendency in tablets.

From the various results, it is notable that the pregelatinized starches had lower P_y but higher P_k values than the native starches. Odeku and Itiola (16) have shown that while P_y relates essentially to the onset of plastic deformation during compression, P_k relates to the amount of plastic deformation occurring during the compression process. From the present results, it would appear that pregelatinization of the starches facilitated faster onset of plastic deformation but reduced the total amount of plastic deformation which occurred during the compression process. This is probably responsible for the higher tensile strength values for the native starches, since higher total plastic deformation would lead to more contact points for interparticulate bonding (14).

CONCLUSION

The results of the present work provide some insight into the compressional characteristics of native and pregelatinized forms of sorghum, plantain, and corn starches and the mechanical properties of their tablets. The results show that pregelatinization increases the densification of the starches during die filling and at low pressures. Pregelatinization also facilitates faster onset of plastic deformation in the starches, but appears to reduce the amount of plastic deformation which occurs during the compression process. Tablets made from pregelatinized starches have lower values of tensile strength and brittle fracture index than those made from native starches.

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