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Original Article

Compaction Properties of Three Types of Starch

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Abstract

A study has been made of the compaction properties of two experimental starches, namely yam starch obtained from *Dioscorea rotundata* and rice starch obtained from *Oryza sativa* and the mechanical properties of their tablets, in comparison with those of official corn starch. The influences of the physical and geometric properties of the starch particles on the compression properties of the starches were determined. Further analysis of the compaction properties was done using density measurements, and the Heckel and Kawakita equations. The mean particle diameter and the shape factor of the starches were directly related to the loose initial density (D_o) and rearrangement at low pressures (D_A) characteristics of the starches, but inversely related to the rearrangement of the particles in the early stages of compression (D_B). The packed initial relative density (D_I) was also directly related to the particle diameter and shape factor. The three starches deform mainly by plastic flow. The values of the mean yield pressures, which are inversely related to the onset of plastic deformation in the starches were in the rank order rice>corn>yam. Another pressure term, inversely related to the amount of plastic deformation during compression, was in the rank order yam>corn>rice. The ranking of the values of both the tensile strength and brittle fracture index of tablets made from the starches was rice>corn>yam, indicating that rice starch tablets exhibited the highest bond strength and brittleness while yam starch exhibited the lowest values. The compaction properties of the experimental starches as characterized compared well with those of official corn starch. The results obtained showed that in tablet formulations, yam starch would be more useful in minimizing the problems of lamination and capping while rice starch would be more useful when high bond strength of the tablet is desired. Thus, yam and rice starches could be useful in formulations to produce tablets with desired mechanical properties for particular purposes.

Keywords: Starch; Corn; Yam; Rice; Compaction properties; Mechanical properties.

Introduction

Starches are used as fillers, binders, disintegrants and lubricants in tablet formulations due to their suitable physicochemical properties and relative inertness. In recent times, a lot of efforts have been made to develop some local starch from different botanical sources as pharmaceutical excipients in tablet formulations (1-5). Yam and rice starches have been found to be useful in the dual role of binder and disintegrant in particular tablet formulations (4). It is however important to characterize the compaction properties of these starches in order to determine the optimum conditions to which they should be subjected during tableting process.

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The compression properties of pharmaceutical powders have been characterized using density measurements, and the Heckel and Kawakita equations (6-8), while the mechanical properties of tablets have been assessed using the tensile strength (T). This is a measure of bond strength and brittle fracture index (BFI) values, being a measure of the brittleness of tablets (9-12).

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:

$$\ln [1/(1-D)] = KP + A \qquad (1)$$

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 A the intercept, the relative density D_A, can be calculated using the following equation (13);

$$D_A = 1 - e^{-A} \qquad (2)$$

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:

$$D_B = D_A - D_o \qquad (3)$$

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

$$C = (V_o - V_p) / V_o = abP / (1 + bP) \qquad (4)$$

The equation, in practice can be rearranged to give:

$$P/C = P / a + 1 / ab \qquad (5)$$

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 Pk which is the pressure required to reduce the powder bed by 50% (14, 15).

The brittle fracture index (BFI) was devised by Hiestand et al (16) and it is obtained by comparing the tensile strength of tablets with a hole at their centre, which acts as a built-in stress concentration defect, with the tensile strength of tablets without a hole, both at the same relative density (9, 16). A low value of BFI is desirable for the minimization of lamination and capping during tablet production. On the other hand, the desirable effect of tensile strength depends largely on the intended use of the tablets (10).

In the present work, a study has been made of the compaction properties of two experimental starches-yam starch obtained from *Dioscorea rotundata* and rice starch obtained from *Oryza sativa* - and the mechanical properties of their tablets, in comparison with those of official corn starch. The effects of the physical and geometric properties of the starches on the compaction properties were also investigated.

Experimental

Materials

The materials used were corn starch BP (BDH, Chemicals Ltd., Poole, U.K), yam starch obtained from *Dioscorea rotundata* and rice starch obtained from *Oryza sativa*. The experimental starches were prepared in our laboratory. The description of the preparation and purification of the starches has been given elsewhere (4, 17). The laboratory starches were of the same purity as industrial starches (>99.8%)

Methods

Characterisation of starches

The size distributions (and shapes) of the starches were determined by optical microscopy on approximately 300 particles picked randomly in the optical field for each starch, from which the values of the mean projected particle diameter (đ) were calculated (10). The true densities were determined by the pycnometer method with xylene as the displacement fluid.

The shape coefficient, α, for all the starches were calculated from the expression (18):

$$\alpha = \rho_s \times Sw \times de + N \qquad (6)$$

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 (15).

The specific surface area of the starches, S_w ($m^2\ g^{-1}$), was calculated using the expression (19):

$$S_w = \alpha_{sv.t} \times S_v / \rho_s \qquad (7)$$

where $\alpha_{sv.t}$ is the surface-volume sieve shape factor, which was taken as 6, i.e the shape factor for a sphere (19), 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}.

Determination of pre-compression density

The bulk density of each starch at zero pressure (loose density) was determined by pouring 20g of the starches at an angle of 45° through a funnel into a glass measuring cylinder with a diameter of 21mm and a volume of 50 mL (20, 21). Determinations were done in triplicate. The relative density D_o, of each starch was obtained from the ratio of its loose density to its particle density.

Preparation of tablets

Quantities (600 mg) of each starch powder, giving tablet thickness of 4.54 ± 0.11 mm at zero porosity as calculated from particle density values, were compressed for 30 seconds with predetermined loads on a Carver hydraulic hand press (Model C, Carver Inc., Menomonee Falls, Wisconsin, U.S.A) 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). Tablets with a hole (1.59mm diameter) at their centre were made using an upper punch with a hole through the centre and a lower punch fitted with a pin (8, 21). After ejection, the tablets were stored over silica gel for 24 h to allow for elastic recovery and hardening, and prevent falsely low yield values. Their weights (w) and dimensions were then determined to within ± 1mg and 0.01 mm respectively, and their relative densities (D).

were calculated using the equation:

$$D = w / V_{t.\rho s} \qquad (8)$$

where V_t is the volume (cm^3) of the tablet (including the hole when present) and ρ_s is the particle density (gcm^{-3}) of the solid material.

Heckel plots of ln (1/1-D) versus applied pressure (P) and Kawakita plots of P/C versus P, were constructed for all the starches.

Testing

The tensile strengths of the normal tablets (T) and apparent tensile strengths of those containing a hole (T_o), were determined at room temperature by diametral compression (22) using an PTB 301 hardness tester (Pharmatest, Switzerland) and by applying the equation:

$$T \text{ (or } T_o) = 2F / (\pi \times dt) \qquad (9)$$

where T (or To) is the tensile strength of the tablet (MNm^{-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 or more and the results given are the means of several determinations.

The Brittle Fracture Index (BFI) of the tablets was calculated using the following equation.

$$BFI = [(T / T_o) - 1] \qquad (10)$$

Statistical analysis

Statistical analysis was carried out to compare the properties of the experimental starches with those of official corn starch using the Analysis of Variance (ANOVA) on a computer software GraphPad Prism ® 4 (GraphPad Software Inc. San Diego, USA). Tukey-Kramer multiple comparison tests was used to compare the individual differences between the starches. At 95% confidence interval, *p* values less than or equal to 0.05 were considered significant.

Results and Discussion

The photomicrographs of the starches as viewed under the light microscope are presented in Figure 1. The size distribution of the starches was 2.5-35 µm, 5-45 µm and 2-30 µm for yam, rice and corn starch respectively. The results of the physical and geometric properties of the starches are presented in Table 1. The rank order of the mean particle diameter (đ), Heywood diameter (de) and the shape factor (α) was yam>rice>corn. The shape factor serves as a proportionality constant between the particle diameter and the particle surface area (23). Thus corn starch which was round and more regular in shape had the lowest shape factor. But as the shape becomes more angular in rice starch, which was polygonal, and yam starch, which was ovoid, the shape factor increased. The specific surface area S_w, is used to describe the area of contact between particles. The larger the specific surface area, S_w, and the smaller the particle size, the larger the area of contact between the particles (20). This observation was true for corn and rice starches. Corn starch which possessed the smallest particle size also had a higher S_w value, while rice starch which had the highest particle size had higher Sw value, while rice starch which had the higher particle size showed the lowest S_w value probably due to its polygonal shape. Usually, the more irregular the particle size of a material, the less the specific surface area (20). On the other hand, yam starch showed the highest mean diameter and also the highest S_w value. This could be due to its particle size, particle size distribution and particle shape.

Figure 2 shows the Heckel plots for the three starches. A linear relationship was obtained with the correlation coefficient of $\geq$0.990 indicating deformation mainly by plastic flow. The values of the mean yield pressure, P_y, D_o, D_A, and D_B for the starches are presented in Table 2. The ranking for P_y was rice>corn>yam; for D_o and D_A, yam>rice>corn and for D_B, corn>rice>yam.

The value of D_o represents the degree of initial packing in the die as a result of die filling for the starches. The D_o was directly related to the shape factor with yam starch showing the highest values and corn starch showing the lowest values. The particles of corn starch are small and thus there are more mechanical and electrostatic forces to prevent packing (20). But as the particle size increases, the electrostatic forces that could prevent closer packing of the particles will decrease.

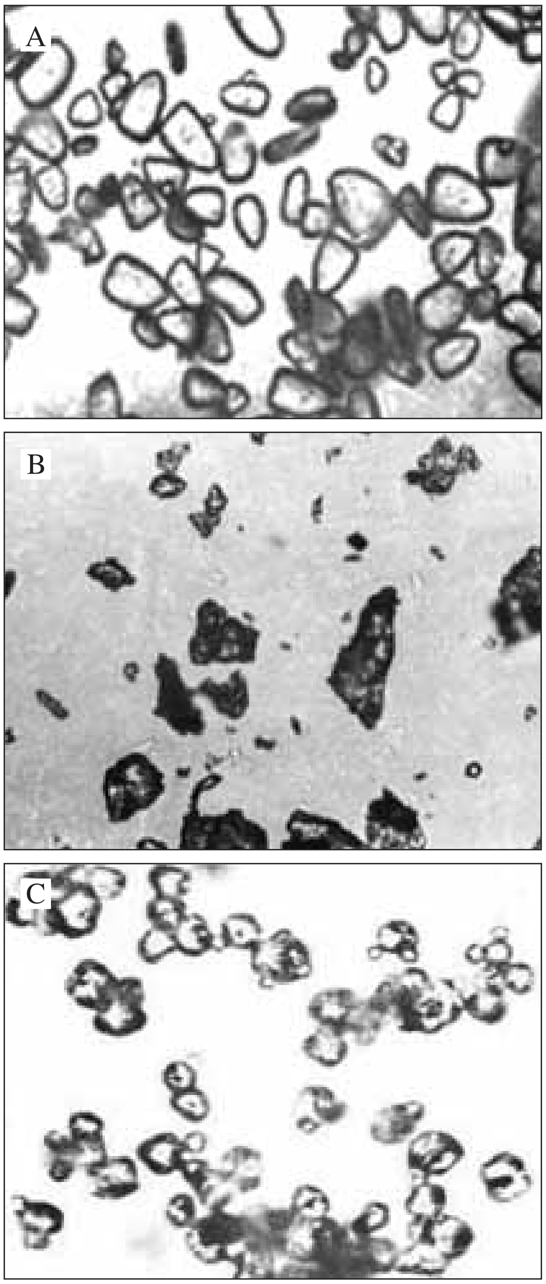

Figure 1. Photomicrographs of the starches. A, yam (mag. X 40); B, rice (mag. X 40); C, corn (mag. X 40).

The value of D_A, which represents the total degree of packing at zero and low pressures, was also found to be directly related to the shape factor. Corn starch, which is spherical in shape and lowest shape factor, had the lowest D_A value while yam starch had the highest value. The

Table 1. Physical and geometric properties of the various starches.

Starch	Geometric shape	đ (μm)	ρ (g.cm^{-3})	de	N	Sw (m^{2}g^{-1})	α
Yam	Ovoid	18	1.475	11.56	1.48	0.049	2.32
Rice	Polygonal	14	1.530	9.88	1.52	0.027	1.93
Corn	Round	8.8	1.479	7.08	1.04	0.031	1.36

values of D_A decreased as the particle shape deviated from spherical (23).

D_B represents the phase of rearrangement of the particles in the early stages of compression. The values of D_B are inversely proportional to the shape factor with corn starch having the highest value and rice starch the lowest. The rearrangement of particles occurs more with smaller particle size (corn) and less with larger and irregular particles (rice and yam starches).

The mean yield pressure P_y is inversely related to the ability of the starches to deform plastically under pressure. The results indicate that yam starch exhibits the fastest onset of plastic deformation during compression while rice starch exhibits the slowest rate. This indicates that yam starch is soft and ductile and readily deforms plastically during compression.

Figure 3 shows the Kawakita plots for all 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 -*a* gives the initial relative density of the starches D_I, while P_K values were obtained from the reciprocal of the values of *b*. The values of D_I and P_K are included in Table 2.

The values of D_I, which is a measure of the packed initial relative density of the starches with the application of small pressures or tapping (11) was in the rank order yam>rice>corn. These values were also seen to be higher than the values of loose initial relative density, D_o. This is in agreement with previous work (11).

The values of P_k, which is an inverse measure of the amount of plastic deformation occurring during the compression process was in the rank order yam>corn>rice. It has been established that the lower the P_k value, the more the total plastic deformation occurring during compression (11). This indicates that yam starch exhibited the lowest amounts of plastic deformation, while rice starch showed the highest amount of plastic deformation during the compression process.

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

$$\text{Log } T \text{ (or } T_o) = AD + B \qquad (10)$$

with a correlation coefficient >0.960. A and B were constants for each starch and depended on whether the tablet had a hole in it or not. Figure 4 shows plots of log tensile strength versus relative density for the starches. The tensile strength of tablet with hole is lower than that of the same without a hole, the hole acting as a stress concentrator (17). Values of T and BFI for the starches at relative density of 0.90, which is representative of commercial tablets, are presented in Table 3. The ranking for T and BFI was rice>corn>yam. Statistical analysis using ANOVA showed that there was statistically significant difference (p<0.001) in the T and BFI values of the various starches. Thus, rice starch exhibited significantly (p<0.001) higher tensile strength and BFI values than corn starch while yam starch exhibited significantly (p<0.001)

Table 2. Parameters derived from density measurements and from Heckel and Kawakita plots.

Starch	Heckel plots				Kawakita plots	
	D_o	P_y (MNm^{-2})	D_A	D_B	P_k (MNm^{-2})	D_I (1 – a)
Yam	0.361	211.01	0.797	0.436	5.655	0.363
Rice	0.348	391.64	0.788	0.413	1.000	0.357
Corn	0.248	254.37	0.746	0.498	1.396	0.290

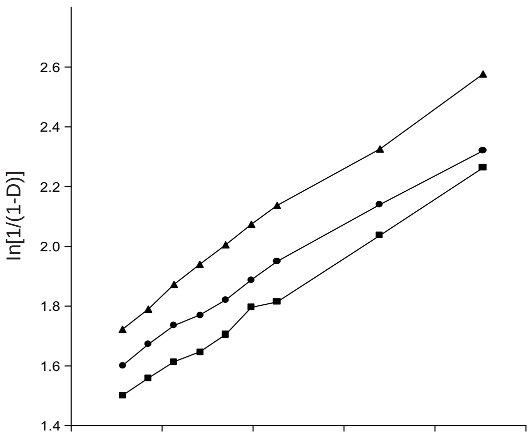

Figure 2. Heckel plots for the starches. ▲, yam; ●, rice; ■, corn.

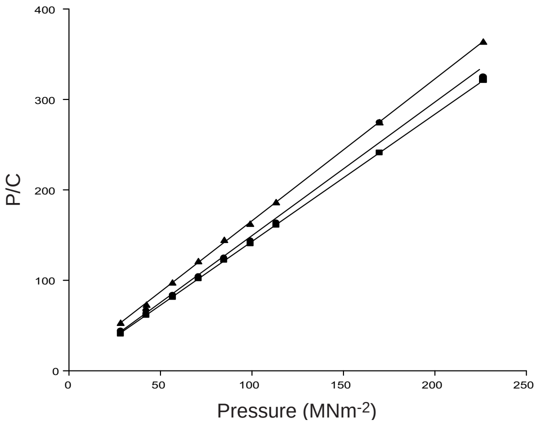

Figure 3. Kawakita plots for the starches. ▲, yam; ●, rice; ■, corn.

lower values of T and BFI than corn starch. Obviously, a low value of BFI is desirable 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 (10). In tablet formulations therefore, yam starch would be more useful in minimizing the problems of lamination and capping while rice starch would be more useful when high bond strength of the tablet is desired.

A comparison of the results on compressional characteristics (Py and Pk) and the bond strength of the starches (T) is noteworthy. Odeku and Itiola (11) have shown that P_y relates essentially to the onset of plastic deformation during compression, while P_k relates to the amount of plastic deformation occurring during the compression process. It is also notable that yam starch which showed the fastest onset of plastic deformation also showed the lowest amount of plastic deformation. On the other hand, rice starch which showed the slowest onset showed the highest amount of plastic deformation during compression. This is probably responsible for the higher tensile strength (T) values of rice starch since higher amount of plastic deformation would lead to more contact points for interparticulate bonding (9).

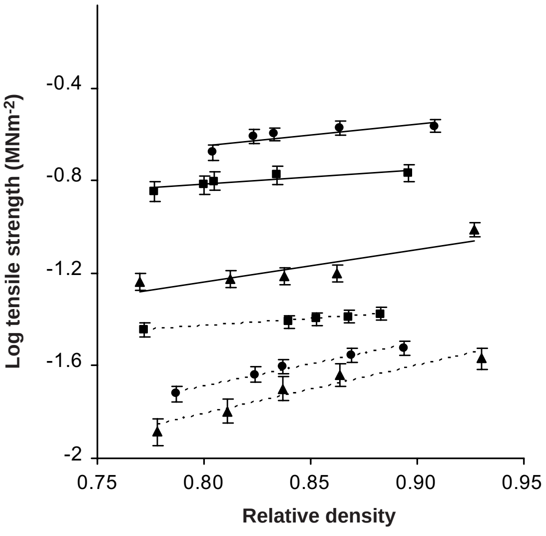

Figure 4. Log tensile strength versus relative density for the starches with (--------) and without (———) a hole at their centre for the starches (mean ± SD, n = 3). ▲, yam; ●, rice; ■, corn.

The results of the present work show that yam and rice starches produced locally deform mainly by plastic flow as official corn starch. The results also showed that in tablet formulations, yam starch would be more useful in minimizing the problems of lamination and capping especially on high speed tabletting machine with short dwell time for the plastic deformation of materials while rice starch would be more useful

Table 3. Tensile strength (T) and Brittle Fracture Index (BFI) values for the various starches at relative density of 0.90 (mean ± SD, n = 3).

Starch	T (MNm^{-2})	BFI
Yam	0.065 (0.005)	0.800 (0.004)
Rice	0.435 (0.026)	5.542 (0.028)
Corn	0.173 (0.012)	1.513 (0.062)

when high bond strength of the tablet is desired. Thus, yam and rice starches could be useful in formulations to produce tablets with desired mechanical properties for particular purposes.

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