Compression and Mechanical Properties of Tablet Formulations Containing Corn, Sweet Potato, and Cocoyam Starches as Binders

O.A. Odeku*, O.O. Awe, B. Popoola, M.A. Odeniyi, and O.A. Itiola

The authors study the effects of sweet potato and cocoyam starches on the compressional and mechanical characteristics of a paracetamol tablet formulation using cornstarch BP.

Starch is one of the most widely used excipients in the manufacture of solid dosage forms. Researchers have tried to develop botanical starches for use as binders and disintegrants in tablet formulations (1–5). For example, sweet potato and cocoyam starches have shown potential as binding agents and/or disintegrants in tablet formulations (3, 6). The effects of these starches on compressional characteristics of the tablets and mechanical properties, however, have not been investigated.

In this article, the effects of sweet potato and cocoyam starches on a paracetamol tablet formulation's compressional characteristics and mechanical properties are compared with those of official cornstarch BP grade. The tablets' compressional characteristics were studied using density measurements and the Heckel and Kawakita equations (7–9). The mechanical properties were assessed using the tensile strength ($T$) (i.e., a measure of bond strength) and the brittle fracture index (BFI) values (i.e., a measure of the brittleness of the tablets) (10–13).

The Heckel equation is widely used for relating a powder bed’s relative density ($D$) during compression to the applied pressure ($P$). The equation is written as follows:

$$\ln \left[ \frac{1}{D} - 1 \right] = KP + A$$

The slope of the straight line portion ($K$) is the reciprocal of the material’s mean yield pressure ($P_y$). From the value of the intercept $A$, the relative density ($D_A$) can be calculated using the following equation (14):

$$D_A = 1 - e^{-A}$$

The powder’s relative density at the point at which the applied pressure equals zero ($D_0$) describes the initial rearrangement phase of densification as a result of die filling. The relative density $D_0$ describes the phase of rearrangement at low pressures and is the difference between $D_A$ and $D_0$:

$$D_0 = D_A - D_0$$

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

$$C = \frac{V_0 - V_p}{V_0} = abP \div (1 + bP)$$

The equation, in practice, can be rearranged as:

$$P \div C = (P \div a) + (1 \div ab)$$

O.A. Odeku is a senior lecturer, O.O. Awe is a research student, B. Popoola is a research student, M.A. Odeniyi is a lecturer, and O.A. Itiola is a professor, all at the Department of Pharmaceutics and Industrial Pharmacy, University of Ibadan, Ibadan, Nigeria. tel. +23 48033235828, pejuodeku@yahoo.com.

*To whom all correspondence should be addressed.
in which $V_0$ is the powder’s initial bulk volume and $V_p$ is the bulk volume after compression. The constant $a$ is the material’s minimum porosity before compression; the constant $b$ relates to the material’s plasticity. The reciprocal of $b$ defines a pressure term $P_b$, which is the pressure required to reduce the powder bed by 50% (15, 16).

The BFI was devised by Hiestand et al. (17) and is obtained by comparing the tensile strengths of tablets with and without a hole at their centers at the same relative density (10, 17). The hole acts as a built-in stress concentration defect.

Paracetamol was chosen because of its poor compression properties. Hence, it needs a binding agent to form satisfactory tablets among other excipients.

### Materials and methods

**Materials.** The materials used were paracetamol BP (Rhone-Poulenc Sante, Paris, France), lactose (DMV, Veghel, Netherlands), cornstarch BP (BDH, Chemicals Ltd., Poole, UK), sweet potato starch obtained locally from the tubers of *Ipomoea batatas*, and cocoyam starch obtained locally from the tubers of *Colocasia esculenta*. The experimental starches were prepared in a University of Ibadan (Ibadan, Nigeria) laboratory. The description of the starches’ preparation and purification has been given elsewhere (2, 4).

**Preparation of granules.** Batches (250 g) of a basic paracetamol–lactose formulation (80:20% w/w) were dry-mixed for 5 min in a planetary mixer (Kenwood Corp., Tokyo, Japan). The mixture was moistened with 40 mL of distilled water or the appropriate amounts of starch mucilage to produce granules containing various concentrations of the starch as binders. Massing continued for 5 min. Then, the wet masses were granulated manually by passing them through a 12-mesh sieve (1400 µm), dried in a hot-air oven for 18 h at 50 °C, and resieved through a 16-mesh sieve (1000 µm). The granules were stored in airtight containers.

**Determination of particle density.** The samples’ particle densities were determined with a pycnometer with xylene as the displacement fluid. An empty 50-mL pycnometer bottle was weighed ($W_1$), filled with xylene (nonsolvent), and the excess wiped off. The filled bottle was weighed a second time ($W_2$). The difference between this weight and $W$ was calculated ($W_2$). A 2-g quantity of the sample was weighed ($W_3$) and quantitatively transferred into the pycnometer bottle. The excess solvent was wiped off and the bottle was weighed again ($W_4$). The particle density, $\rho_p$ (g/cm³), was calculated from the following equation:

$$\rho_p = \left(\frac{W_1 \times W_2}{W_2} + 50(W_4 - W_2 + W_3) + W\right) \text{[6]}$$

**Determination of precompression density.** The bulk density of each formulation at zero pressure (loose density) was determined by pouring the granules at a 45° angle through a funnel into a 50-mL glass measuring cylinder with a diameter of 21 mm (2, 18). Determinations were conducted in triplicate. The relative density ($D_T$) of each formulation was obtained from the ratio of its loose density to its particle density.

**Preparation of tablets.** Tablets (500 mg) were prepared from the 500–1000-µm granules by compressing them for 30 s with predetermined loads on a hydraulic hand press (model C. Carver Inc., Menomonee Falls, WI). Before each compression, the 10.5-mm die and flat-faced punches were lubricated with a 2% w/v dispersion of magnesium stearate in an ether–ethanol (1:1) solution. Tablets with a hole (1.59-mm diameter) at their centers were made using an upper punch with a hole and a lower punch with a pin (2, 10). After ejection, the tablets were stored over silica gel for 24 h to allow for elastic recovery and hardening. This procedure prevents false low-yield values.

The tablets’ weights and dimensions were determined to within ±1 mg and 0.01 mm, respectively. Their relative densities ($D$) were calculated using the equation:

$$D = \frac{W}{W \times \rho_p} \text{[7]}$$

in which $V_p$ is the tablet’s volume (cm³), including the hole when present, and $\rho_p$ is the solid material’s particle density (g/cm³). Heckel plots of $\ln (1 + \frac{P}{P_0})$ versus applied pressure ($P$) and Kawakita plots of $P = C + P_0$ versus $P$ were made for all formulations.

**Testing.** The tensile strengths of the normal tablets ($T_0$) were determined at room temperature by diametral compression (19) using a hardness tester (PTB 301, Pharmatest, Switzerland) and by applying the equation:

$$T (or T_0) = 2F + \pi dt \text{[8]}$$

in which $T$ (or $T_0$) is the tensile strength of the tablet (MN/m²), $F$ 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 that split cleanly into two halves without any

### Table I: Parameters derived from density measurements and from Heckel and Kawakita plots.

<table>
<thead>
<tr>
<th>Starch</th>
<th>Sample concen. (% w/w)</th>
<th>$D_T$</th>
<th>$P_T$ (MN/m²)</th>
<th>$D_T$</th>
<th>$P_T$ (MN/m)</th>
<th>$D_T$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sweet potato</td>
<td>0.00</td>
<td>0.228</td>
<td>176.03</td>
<td>0.736</td>
<td>0.508</td>
<td>4.066</td>
</tr>
<tr>
<td></td>
<td>2.50</td>
<td>0.230</td>
<td>360.29</td>
<td>0.831</td>
<td>0.601</td>
<td>2.565</td>
</tr>
<tr>
<td></td>
<td>5.00</td>
<td>0.248</td>
<td>276.45</td>
<td>0.823</td>
<td>0.575</td>
<td>2.462</td>
</tr>
<tr>
<td></td>
<td>7.50</td>
<td>0.257</td>
<td>239.75</td>
<td>0.822</td>
<td>0.565</td>
<td>2.311</td>
</tr>
<tr>
<td></td>
<td>10.00</td>
<td>0.263</td>
<td>217.12</td>
<td>0.817</td>
<td>0.554</td>
<td>2.121</td>
</tr>
<tr>
<td>Cocoyam</td>
<td>2.50</td>
<td>0.222</td>
<td>270.01</td>
<td>0.849</td>
<td>0.627</td>
<td>2.627</td>
</tr>
<tr>
<td></td>
<td>5.00</td>
<td>0.242</td>
<td>219.35</td>
<td>0.848</td>
<td>0.606</td>
<td>2.196</td>
</tr>
<tr>
<td></td>
<td>7.50</td>
<td>0.254</td>
<td>183.38</td>
<td>0.841</td>
<td>0.587</td>
<td>2.014</td>
</tr>
<tr>
<td></td>
<td>10.00</td>
<td>0.261</td>
<td>164.30</td>
<td>0.831</td>
<td>0.570</td>
<td>1.862</td>
</tr>
<tr>
<td>Corn</td>
<td>2.50</td>
<td>0.215</td>
<td>229.60</td>
<td>0.811</td>
<td>0.596</td>
<td>4.966</td>
</tr>
<tr>
<td></td>
<td>5.00</td>
<td>0.234</td>
<td>197.06</td>
<td>0.799</td>
<td>0.565</td>
<td>4.272</td>
</tr>
<tr>
<td></td>
<td>7.50</td>
<td>0.248</td>
<td>187.86</td>
<td>0.795</td>
<td>0.547</td>
<td>3.875</td>
</tr>
<tr>
<td></td>
<td>10.00</td>
<td>0.258</td>
<td>157.57</td>
<td>0.770</td>
<td>0.512</td>
<td>3.192</td>
</tr>
</tbody>
</table>

**Abbreviations:** $D_0$ is the relative density at zero pressure; $P_T$ is the mean yield pressure; $D_T$ is the relative density from the value of intercept $A$; $D_T$ describes the phase of rearrangement at low pressures ($D_T$ – $D_T$); $P_T$ is the pressure required to reduce powder bed by 50% (reciprocal of $b$); and $D_T$ is the initial relative density.
sign of lamination. All measurements were made in triplicate or more. The results given are the means of several determinations.

The tablets’ BFI were calculated using the following equation:

\[ BFI = \left( \frac{(T \div T_0) - 1}{T_0} \right) \]  \[ \text{[9]} \]

**Results and discussion**

Figure 1 shows representative Heckel plots for paracetamol formulations containing 5% of the starches as a binder. \( P_v \) was calculated from the regions of the plots showing the highest correlation coefficient of \( \geq 0.990 \) for all the formulations (usually 84.93–226.47 MN/m²). The intercept \( A \) was determined from the extrapolation of the line. The values of the mean yield pressure, \( D_0, P_s, D_0, \) and \( D_s \) for the formulations are presented in Table I. The \( D_0 \) value—which represents the degree of initial packing in the die as a result of die filling—increased as the starch concentration increased. Formulations containing a sweet potato binder had the highest \( D_0 \) values; formulations containing cornstarch had the lowest. This result indicates that formulations containing sweet potato starch exhibited the highest degree of packing in the die because of die filling; formulations containing cornstarch exhibited the lowest values.

The \( D_0 \) values, which represent the total degree of packing at zero and low pressures, decreased as the starch concentration increased. In general, formulations containing cornstarch showed the lowest \( D_0 \) values; those containing cocoyam starches exhibited the highest values.

The \( D_0 \) value represents the particle rearrangement phase in the early compression stages and tends to indicate the extent of particle or granule fragmentation, although fragmentation can occur concurrently with plastic and elastic deformation of constituent particles. The \( D_0 \) value also decreased with an increase in starch content. Formulations containing cocoyam starch exhibited the highest values; those containing cornstarch showed the lowest values. This result indicates that granule fragmentation decreased with an increase in starch concentration. Furthermore, the values of \( D_0 \) were usually higher than those of \( D_s \) because granule fragmentation and the subsequent filling of void spaces between particles occurred extensively at low pressures. The loose packing of the large granules at zero pressure tended to yield low \( D_s \) values (10).

The mean yield pressure, \( P_s \), is inversely related to the formulations’ ability to deform plastically under pressure. The \( P_s \) values decreased with an increase in the starch concentration. Formulations containing sweet potato starch exhibited the highest \( P_s \) values, and those containing cornstarch exhibited the lowest \( P_s \) values. These results indicate that formulations containing cornstarch as a binder exhibited the fastest onset of plastic deformation during compression. Formulations containing sweet potato exhibited the slowest rate. This finding indicates that cornstarch plastically deforms faster during compression than the experimental starches.

Figure 2 shows representative Kawakita plots for paracetamol formulations containing 5% w/w of the starches as a binding agent. A linear relationship was obtained at all compression pressures used with a 0.999 correlation coefficient for all starches. Values of \( a \) and \( ab \) were obtained from the slope and intercept of the plots, respectively. Values of \( 1-a \) yield the initial relative density of the starches (\( D_0 \)) while \( P_s \) values were obtained from the reciprocal of \( b \) (see Table I).

The \( D_0 \) values, which is a measurement of the packed initial relative density of the starches with the application of small pressures or tapping (12), decreased as the concentration of starches increased. These values are also higher than the corresponding values of loose initial relative density, \( D_0 \). This result corresponds with previous findings of Odeku and Itiola (12).

The \( P_s \) values, which are an inverse measurement of the plastic deformation occurring during the compression process (12), also decreased with an increase in the starch concentration. In general, \( P_s \) values rankings (i.e., the order of magnitude of the various values for the relevant parameters) for the formulations containing the various starches was cocoyam, followed by sweet potato, and then corn. Thus, formulations containing cornstarch exhibited the lowest degree of total plastic deformation during the compression process, and formulations containing cocoyam starch exhibited the highest values.

It has been established that the lower the \( P_s \) value, the more the total plastic deformation occurs during compression (12). Although formulations containing cornstarch exhibited a faster onset of plastic deformation during compression as indicated by the low \( P_s \) values, they also exhibited the lowest degree of plastic deformation during the compression process. On the other hand, formulations containing the experimental starches exhibited a slower onset of plastic deformation but a higher amount of plastic deformation during compression. Thus, cornstarch will be more useful than other experimental starches in a high-speed tablet machine with a short dwell time.

The results of the tensile tests on the paracetamol tablets fit the general equation (correlation coefficient > 0.980):

\[ \log(T \text{ or } T_0) = aD + b \]  \[ \text{[10]} \]
in which $a$ and $b$ were constants for each formulation but depended on whether the tablet had a hole in it. Figure 4 shows representative plots of log tensile strength versus relative density for formulations containing 5% w/w of the starches as binder. The tensile strength of tablets with a hole is lower than those without a hole (17). Table II presents the values of $T$ and BFI for the starches at a relative density of 0.90, which represents commercial paracetamol tablets.

The tensile strength of the paracetamol tablets increased, but the BFI decreased with an increase in starch concentration. Formulations containing cocoyam starch, which frequently exhibited the highest tensile strength, also had the highest BFI. Formulations containing cornstarch exhibited the lowest $T$ and BFI values. A low BFI is desirable for minimal lamination and capping during tablet production, but the effect on tensile strength largely depends on the intended use of the tablets (12). Furthermore, the low $P_k$ values for tablets containing the experimental starches is probably responsible for the tablets’ higher $T$ values because greater total plastic deformation creates more contact points for interparticulate bonding (5, 10).

Therefore, cornstarch would be more useful than the experimental starches for minimizing the problems of lamination and capping, especially on high-speed tableting machines with short dwell times for the plastic deformation of materials. Cocoyam and sweet potato starches would be useful when a tablet’s high bond strength is needed.

**Conclusion**

This article provides insight into the effects of the experimental starches on paracetamol tablet formulation’s compressional characteristics and mechanical properties. The results indicate botanical starches could be useful to produce tablets with desired mechanical properties for specific purposes depending on whether stronger or softer tablets are required in cases such as chewable or disintegrating tablets.

**Acknowledgement**

Part of this work was supported by a Senate research grant from the University of Ibadan.

<table>
<thead>
<tr>
<th>Starch</th>
<th>Sample concen. (%) w/w</th>
<th>$T$ (MN/m²)</th>
<th>BFI</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sweet potato</td>
<td>0.00</td>
<td>0.505</td>
<td>0.011</td>
</tr>
<tr>
<td></td>
<td>2.50</td>
<td>0.891</td>
<td>0.529</td>
</tr>
<tr>
<td></td>
<td>5.00</td>
<td>1.122</td>
<td>0.397</td>
</tr>
<tr>
<td></td>
<td>7.50</td>
<td>1.667</td>
<td>0.265</td>
</tr>
<tr>
<td></td>
<td>10.00</td>
<td>1.933</td>
<td>0.259</td>
</tr>
<tr>
<td>Cocoyam</td>
<td>2.50</td>
<td>0.953</td>
<td>0.619</td>
</tr>
<tr>
<td></td>
<td>5.00</td>
<td>1.231</td>
<td>0.535</td>
</tr>
<tr>
<td></td>
<td>7.50</td>
<td>1.739</td>
<td>0.429</td>
</tr>
<tr>
<td></td>
<td>10.00</td>
<td>1.988</td>
<td>0.337</td>
</tr>
<tr>
<td>Corn</td>
<td>2.50</td>
<td>0.602</td>
<td>0.250</td>
</tr>
<tr>
<td></td>
<td>5.00</td>
<td>0.862</td>
<td>0.233</td>
</tr>
<tr>
<td></td>
<td>7.50</td>
<td>0.961</td>
<td>0.225</td>
</tr>
<tr>
<td></td>
<td>10.00</td>
<td>1.378</td>
<td>0.191</td>
</tr>
</tbody>
</table>