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Compressional characteristics of four starches

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Compression data about barley, corn, potato and wheat starches were obtained by two methods: the ejected tablet method and the tablet-in-die-method. These data were analysed using the Heckel and the Cooper-Eaton equations. The Heckel equation appeared to be the more sensitive in distinguishing the various stages during the compression. Die filling and rearrangement processes for the starches were especially dependent on particle size and shape and thus on contact area between particles. Densification of large starch particles (potato starch) owed more to die filling and less to rearrangement. Densification of small particles (corn starch) was the reverse. Starch having a wide particle size distribution (wheat) or an irregular particle shape (barley) underwent a relatively small amount of densification as a result of die filling and a relatively great amount of densification because of rearrangement of particles during tableting. The tendency of the starches to total and pure plastic deformation was dependent on particle size, size distribution and particle shape. Corn starch was the most prone to plastic flow with only little elastic recovery. Potato starch also flowed plastically with ease. Barley and wheat starches were the more elastic.

Various authors have distinguished stages during the compression of powders in dies (Huffine & Bonilla 1962; Heckel 1961 a,b; Cooper & Eaton 1962). These include the filling and rearrangement of particles in the die, the elastic and plastic deformation of particles, the fracture of particles and cold working. In many instances several of these processes probably occur simultaneously. Numerous equations have been proposed to describe compressional behaviour of powders to differentiate the various stages in a compression process (Kawakita & Tsutsumi 1966; Kawakita & Ludde 1970/71). Conclusions have been drawn concerning the effect of the characteristics of the material and the particle size on the densification process.

Potato and corn starches are the most commonly used starches for tablets. Wheat and barley starches could be candidates for tablet adjuvants (Sakr et al 1974; Juslin et al 1981; Paronen & Juslin 1981). Owing to the close chemical and physical relationships between different starches it seemed to be worthwhile to evaluate the compressional characteristics of these starches. It has been suggested that starches deform mostly by plastic flow (Führer et al 1975; David & Augsburger 1977; Rees & Rue 1978). The clearest differences between these starches are those of size, size distribution and particle shape.

The purpose of the present work was to see if a relationship existed between the particle characteristics and the compressional behaviour of potato, corn, wheat and barley starches.

MATERIALS AND METHODS

Materials

The starches studied were corn starch (Amylum maydis, Ph.Eur.), potato starch (Amylum solani, Ph.Eur.), wheat starch (Amylum tritici, Ph.Eur.) and barley starch (Amylum hordei, commercial grade, Hämeen Peruna Oy, Jokioinen, Finland). Some particle characteristics of the starches are shown in Table 1.

Table 1. Particle characteristics of the starches.

Starch	d_{av} (μm)	± r.s.d. (%)	d_{sv} (μm)	S_w (m²/g)	F_s (–)
Barley	14·4	48·2	20·5	0·26	7·97
Corn	11·4	49·0	17·7	0·28	7·41
Potato	18·9	52·1	29·4	0·17	7·47
Wheat	13·3	64·1	23·2	0·22	7·63

d_{av} ± r.s.d. = arithmetic mean diameter ± relative standard deviation, d_{sv} = surface-volume diameter, S_w = specific surface area, F_s = shape factor.

Particle characteristics

The particle size distribution of the starches was examined microscopically by measuring the diameter of four hundred particles. Fig. 1 and Table 1 show that the number of small particles is greatest in wheat and barley starches, whilst corn starch has the smallest mean particle size.

The mean surface-volume diameter, d_{sv}, of four hundred particles of the starches was counted from the equation (Allen 1975):

$$d_{sv} = \frac{\Sigma\, nd^3}{\Sigma\, nd^2}$$

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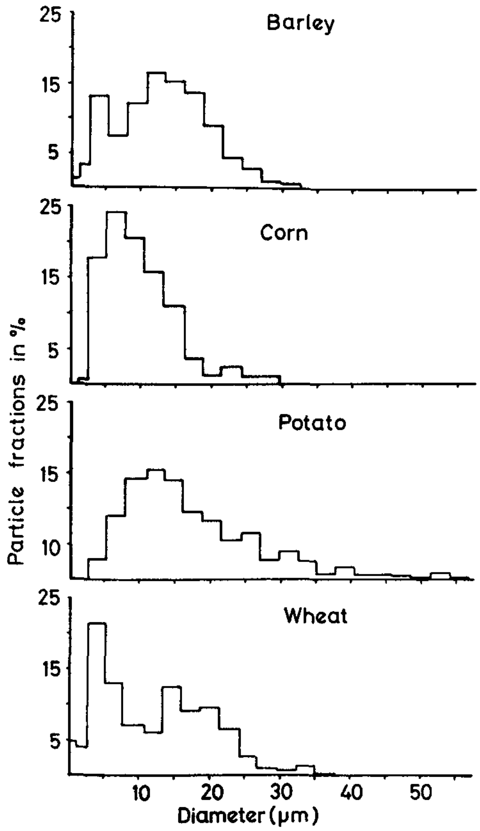

FIG. 1. Particle size distribution of different starches

where n is the number of particles of diameter d. A unit volume of monosized particles with a diameter d_{sv} will have a total surface area identical with the surface of a unit volume of the actual sample having a mean surface-volume diameter d_{sv}.

The specific surface area per unit weight, S_w, of the starches was determined by BET method from the adsorption of krypton gas at the boiling point of liquid nitrogen, using Orr Surface Area, Pore Volume Analyzer, Model 2100E (Micromeritics Instruments Corp.).

The shape of the particles of the starches was studied by means of micrographs taken by Jeol JSM-35 scanning electron microscope, cf. Fig. 2. The particle shape factor F_s (Allen 1975)

$$F_s = S_w \rho_t d_{sv}$$

where ρ_t is the effective particle density, was used to obtain numerical values that describe the shape of the starch particles. The value of shape factor is larger the more irregular the particle shape.

Powder characteristics

The water content of the materials was determined on a Mettler Drying Unit LP 12. The averages of three determinations were used for conclusions. The water content in weight per cent was for corn 9·7, for potato 10·6, for wheat 9·5 and, for barley starch 9·4.

Magnesium stearate, 0·5 weight per cent, was added to the starch powders as a lubricant just before the density and tableting experiments. The loose density, ρ_0, was determined by pouring a quantity of tablet mass through a funnel in a fine stream into a glass measuring cylinder with a diameter of 22 mm and volume of 50 ml. The cylinder was placed at an angle of 45°. Six determinations were made for conclusions. The loose density (g ml^{-1}) was for corn 0·500, for potato 0·707, for wheat 0·504 and, for barley starch 0·437.

The effective density of loosely compressed tablets of the lubricated starch powders was determined with the air comparison pycnometer, Beckman model 930, using helium as the inert gas. Ten determinations were made, and the effective particle density was 1·495 g m^{-1} for all the starches.

Compression of the tablets

As effective particle density was equal for all the starches we were able to use the same amount of them for tableting and still theoretically have the same tablet thickness at infinite pressure.

Tablets were compressed using an instrumented single punch machine (Puumalainen et al 1978). The accuracy of displacement measurements was verified using the methods of Juslin & Paronen (1980). The samples of 550 mg were compressed at a speed of 35 tablets min^{-1}. The contact time of the upper punch with the surface of powder column was about 0·15 s. All the tablets were compressed using the fixed lower punch position, which was the same as during the calibration of displacement measurements.

Two methods were used to find the tableting parameters. Firstly, the applied pressure and the packing fraction of a powder column were determined at at least 20 points during the compression phase. The data from six tablets were used for conclusions by this 'tablet-in-die-method'. Secondly the maximum upper punch pressure and packing fractions were determined by measuring the dimensions of the tablet with a micrometer screw about 24 h after the ejection of the tablet. The data from ten tablets were used for conclusions by this 'ejected tablet method'.

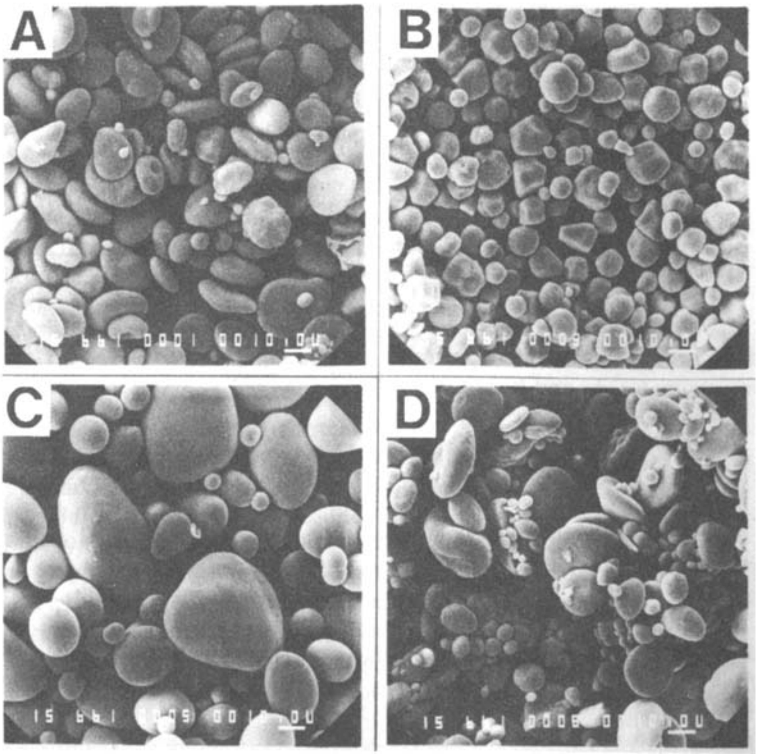

FIG. 2. Scanning electron micrographs of starch powders. A, Barley starch. B, Corn starch. C, Potato starch. D, Wheat starch. Bar = 10 μm.

Compression equations

The relations between the applied pressure and the density of the powder columns were analysed by means of Heckel (1961 a,b) and Cooper-Eaton (1962) equations.

The Heckel equation is

$$\ln(1/(1-D)) = kP + A \qquad (1)$$

where D is the packing fraction of the tablet, or the apparent density of the tablet vs the effective particle density. P is the applied pressure. The constants k and A are determined from the slope and intercept respectively of the extrapolated linear portion of the plot of ln(1/(1–D)) vs P. The constant k, obtained from the ejected tablet method, has been identified with the reciprocal of the yield pressure, K, of the material (Hersey & Rees 1971). Fell & Newton (1971) have pointed out that k, obtained from the tablet-in-die-method, gives a low value to the yield pressure, which describes the tendency of a material to deform by plastic flow or by fragmentation. This is due to the elastic component of this method. The constant A has been identified with the movement of the particles during the initial stages of the compression.

Cooper & Eaton (1962) considered the compaction of powders to take place in two stages: firstly, the filling by particle movement of voids of the same or larger size than the particles, secondly, the filling of the smaller voids by plastic deformation or fragmentation of particles. Their equation is (Kurup & Pilpel 1978)

$$\frac{1/D_0 - 1/D}{1/D_0 - 1} = a_1 e^{-k_1/P} + a_2 e^{-k_2/P} \qquad (2)$$

where D_0 is the packing fraction when the applied pressure equals zero or the loose density of powder vs the effective particle density. a_1 and a_2 are dimensionless coefficents indicating respectively the fraction of the theoretical compaction that could be achieved at infinite pressure by the filling of the voids of the same size and of smaller size than the particles. $a_1 + a_2$ equals unity when the compaction can be completely described in terms of the two separate mechanisms referred to above. A sum greater than unity indicates that an absolutely dense compact can be achieved by these two mechanisms at lower than infinite pressure. If the sum is less than unity, this indicates that other processes are operating before complete compaction is achieved. k_1 and k_2 are identified as the pressures at which the respective processes occur with the greatest probability.

For some materials, e.g. alumina and lactose, the plot of

$$\ln \frac{1/D_0 - 1/D}{1/D_0 - 1} \quad \text{versus } 1/P$$

exhibits a kink, the two linear regions representing consolidation by the two different mechanisms (Kurup & Pilpel 1978; York & Pilpel 1973). The values of a_1 and $a_1 + a_2$ are determined from the intercept of the first and the second linear regions, respectively, on the ordinates. k_1 and k_2 are determined by means of the slopes of these two linear regions.

Evaluation of the tablets

The scanning electron micrographs were of the surfaces of broken tablets compressed at a pressure of about 200 MPa.

RESULTS AND DISCUSSION

Packing fraction in the bulk state

The packing fraction of starch powders, D_0, obtained from the ratio of loose density of powders to effective particle density of the starches, describes the die filling (cf. Table 2). Potato starch forms the densest and barley starch the loosest column in this phase. Potato starch has the greatest mean particle diameter and thus the least electrostatic forces that could prevent the packing of particles in the bulk state. The particles of barley starch are small and irregular and thus there are mechanical and electrostatic forces to prevent packing. According to the values describing particle characteristics and also the value for the packing fraction in the bulk state, wheat starch is intermediate between the other starches. The value for the packing fraction in the bulk state is also intermediate for corn starch. The particles of corn starch are very small but regular in shape. Thus it seems that the values for the packing fraction in the bulk state are strongly dependent both on the particle size and on the particle shape.

Packing fractions in the compression stage

The packing fractions from the Heckel equation are given in Table 2. Heckel plots in Fig. 3 obtained for the starches from the tablet-in-die-method differ from each other. Potato starch becomes more dense in the precompression phase, particularly during the die filling and also in the early stages of downwards movement of the upper punch into the die. Corn starch seems to oppose the densification process most strongly, wheat and barley starches being intermediate in the early stages of the compression compared with potato and corn starches. The plots obtained from the ejected tablet method differ from those obtained from the tablet-in-die-method. The reason is the elastic recovery after ejection of the tablets from the die. The plots show that barley and wheat starches are more elastically deforming materials than corn and potato starches.

Table 2. Packing fractions obtained from density measurements and from Heckel plots.

		Tablet-in-die method		Ejected tablet method	
Starch	D_o	D_A	D_B	D_A	D_B
Barley	0·292	0·612	0·320	0·610	0·318
Corn	0·334	0·571	0·237	0·585	0·251
Potato	0·473	0·638	0·165	0·629	0·156
Wheat	0·337	0·610	0·273	0·621	0·284

D_o = packing fraction of powder.
D_A = densification due to die filling and rearrangement.
D_B = densification due to rearrangement.

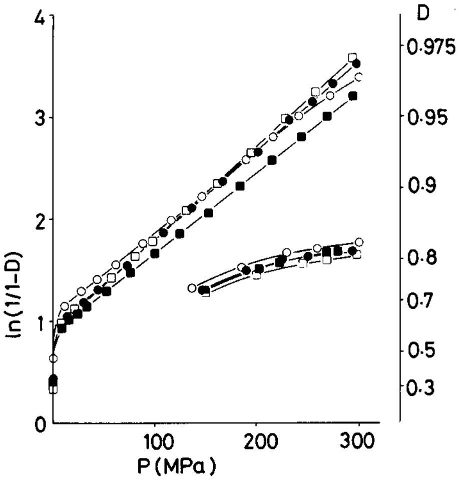

FIG. 3. Graphs of compressional pressure (P) versus ln(1/(1–D)) (=Heckel plots) for four starches obtained either from the tablet-in-die-method (upper four curves) or from the method of ejected tablets (lower four curves). □ = Barley starch ■ = Corn starch ○ = Potato starch ● = Wheat starch.

The data of the mean surface-volume diameter and the specific surface area can be used to describe the contact area between particles. A smaller d_{sv}-value and a larger S_w-value mean a larger surface for contact. The contact area between particles in the powder column becomes larger as the particle size decreases. This is obvious with comparing the values of corn and potato starches, cf. Table 1. Besides particle size, the particle size distribution and particle shape also affect the contact area between particles (Rees 1977). The wide particle size distribution (wheat starch) and the great irregularity of particle shape (barley starch) increase the area for contact between particles. Where the contact area is large, there exists more resistance to particle movement of particles. This is seen from the D_A-values of the starches, which describe the share of densification due to the above processes. D_A-values are obtained from the constant, A, which is the intercept of the linear portion of the Heckel plots in the pressure range 30–250 MPa in the tablet-in-die-method and 135–300 MPa in the ejected tablet method. Differences in the surface properties of the starches, e.g. in hardness of the surfaces, may also have some effect on this phase of compression, but any effect is difficult to distinguish.

It is obvious that the phase of rearrangement of particles is larger for materials with relatively less densification due to die filling. The extent of the rearrangement phase D_B also depends on the theoretical point of densification at which deformation of particles begins. D_B is obtained from the difference D_A-D_0.

The values of D_B support the findings of Huffine & Bonilla (1962) for sodium chloride, and of Fell & Newton (1971) for crystalline and spray-dried lactose, and of York (1978) for four pharmaceutical materials. The rearrangement of particles occurs more with smaller particle size (corn) and less with larger particle size (potato) and seems to be particularly responsible for the densification of wheat and barley starches which have a relatively wide particle size distribution and an irregular particle shape.

No differences of note were found between the use of the tablet-in-die-method and the ejected tablet method in determining the packing fractions by means of the Heckel equation.

The packing fractions of the Cooper-Eaton equation are in Table 3. There is a clear kink in all the Cooper-Eaton plots (Fig. 4) obtained by the tablet-in-die-method, but not in the plots obtained by the ejected tablet method. The reason for this could be that we could not produce unbroken tablets at compression pressures below 100 MPa and thus we were not able to get any value for the Cooper-Eaton equation over the reciprocal pressure of 0·01 MPa^{-1}. Thus the graphs remained practically linear for all the starches. Kurup & Pilpel (1978) have presented an equation for this kind of situation,

$$\ln \frac{1/D_0 - 1/D}{1/D_0 - 1} = \frac{-Q}{P} + \ln R \qquad (3)$$

where ln R is the intercept on the ordinate and Q is the slope. R equals the value of $a_1 + a_2$ from equation 2 and thus the R-value can be used to determine the total fraction of the theoretical compaction achieved by filling voids of the same and smaller dimensions than the particles. Q is not mathematically the same as k_2 obtained from equation 2 but the Q-value can be used as a measure of the hardness and compressibility of the various materials (Kurup & Pilpel 1978).

Table 3. Packing fractions obtained from Cooper-Eaton plots.

Starch	Tablet-in-die method a_1	Tablet-in-die method $a_1 + a_2$	Ejected tablet method R
Barley	0·836	1·012	0·968
Corn	0·781	1·017	0·978
Potato	0·689	1·012	0·969
Wheat	0·807	1·013	0·964

a_1 = fraction of theoretical compaction that would be achieved at infinite pressure for the filling of voids of the same dimensions as the particle.

$a_1 + a_2$ and R = total fraction of theoretical compaction that can be achieved by filling voids of the same and smaller dimensions than the particles.

The packing fractions a_1 obtained from the tablet-in-die-method in the reciprocal pressure range greater than 0·04 MPa^{-1}, correlate well with the results D_B in Table 2 from Heckel plots obtained by

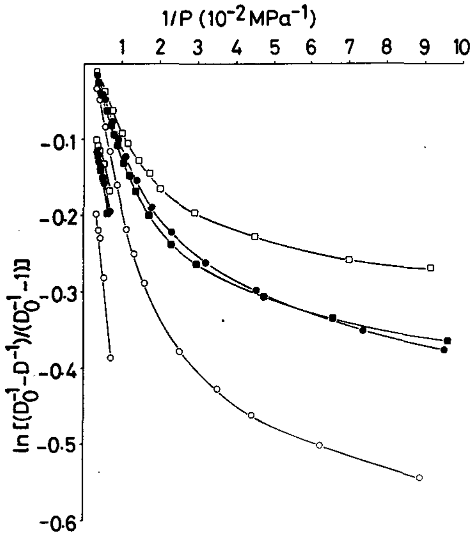

FIG. 4. Graphs of the reciprocal of compressional pressure (1/P) versus $\ln((D_0^{-1} - D^{-1})/(D_0^{-1} - 1))$ (= Cooper-Eaton plots) for four starches obtained either from the tablet-in-die-method (curves) or from the ejected tablet method (straight lines). □ = Barley starch. ■ = Corn starch. ○ = Potato starch. ● = Wheat starch.

the same method. All the values of the packing fraction a_1 from equation 2, which thus describe the densification due to the rearrangement of particles, are larger than values D_B from equation 1. The reason for this is that there is no compaction process (filling large holes) and so a_1-values are only the theoretical upper limit for the packing fraction of the rearrangement (Cooper & Eaton 1962).

The values $a_1 + a_2$ of the tablet-in-die-method from equation 2 in the reciprocal pressure range less than 0·02 MPa^{-1} are greater than unity for all the starches. This indicates that it should be possible to compact all the samples to zero porosity by the two mechanisms described by Cooper & Eaton.

The values R of the ejected tablet method from equation 3 are all below unity. This is because the consolidation caused by the elastic deformation of the starch particles has been eliminated from these values. The limit value of the elasticity is nearly the same for all the starches although there are greater differences in the magnitude of the elasticity in the practical range of compression pressures.

The constants of deformation

The constant k of the Heckel equation was determined from the slope of extrapolated linear portion in the pressure range 135–300 MPa of the plots obtained by the ejected tablet method and in the pressure range 30–250 MPa of the plots obtained by the tablet-in-die-method. The yield pressure of the material has been shown to be the reciprocal of the slope of the Heckel plot obtained by the ejected tablet method (Hersey & Rees 1971). The above yield pressure may be called the yield pressure of plastic deformation, K_p. The yield pressure of total deformation, K_d, is determined by taking the reciprocal of the slope of the plots obtained by the tablet-in-die method. K_d-values also include the effect of the elastic deformation of the particles. The values of K_p and K_d for different starches are shown in Table 4.

If the deformation properties of the starches are considered as nearly identical, there would then seem to be a relationship between the particle properties and the K_d-values of starches. Corn starch has the smallest particle size and its K_d-value is greatest. The mean particle size of potato starch is clearly larger and its K_d-value smaller than that of corn starch. The yield pressures of total deformation for barley starch (irregular particle shape) and especially for wheat starch (wide particle size distribution) are lower than the values for the other two starches. Because a relationship exists between the K_d-value and the particle properties, it seems reasonable to suppose that this value, at least to some extent, depends on the particle properties of the material.

Table 4. Yield pressure and the pressure to fill voids within compacts in MPa.

	Tablet-in-die method			Ejected tablet method	
Starch	K_d	k_1	k_2	K_p	Q
Barley	110	1·0	10·5	411	20·5
Corn	125	1·2	14·3	344	26·8
Potato	120	2·0	20·8	369	48·0
Wheat	109	1·6	12·9	402	23·4

K_d = the yield pressure of total deformation obtained from Heckel plot.

K_p = the yield pressure of plastic deformation obtained from Heckel plot.

k_1 = the pressure necessary to fill the interparticle void space of the greater and same dimensions than the particles obtained from Cooper-Eaton plot.

k_2 and Q = the pressure necessary to fill the interparticle void space of smaller dimensions than the particles obtained from Cooper-Eaton plot.

The above results are in accordance with York's (1978) results from the tablet-in-die-method for four different materials. Fell & Newton (1971) have also shown the dependence of both K_d and also K_p-values on particle size. Their results show that there is a particle size fraction of crystalline and spray-dried lactose that opposes deformation more than other fractions. Hersey et al (1973) also have shown the effect of particle size on the K_p-values of crystalline lactose. The relationship of the yield pressures of plastic deformation, K_p, of the starches is not so close as that seen with the yield pressures of total deformation, K_d. The reason for this is the large and inconstant elastic recovery. The results of Fell & Newton (1971) and Hersey et al (1973) obtained by the ejected tablet method, are not comparable with our results similarly obtained. They used lactose, which undergoes fragmentation of particles with ease but has little elastic deformation (Cole et al 1975; York & Baily 1977). Thus the Heckel plots of lactose obtained by the two methods used will not differ from each other as much as they do for the corresponding plots of the starches. It seems that the tablet-in-die-method distinguishes the effect of the particle size and shape on the general deformation behaviour more clearly than the ejected tablet method, especially when elastic recovery of the material is considerable. The ejected tablet method is, however, the method of choice when only the factors that affect plastic deformation are studied.

The K_p-values of the starches are all high and it would seem that the extensive plastic flow of the particles begins in a lower pressure range. The scanning electron micrographs (cf. Fig. 5) of the surfaces of broken tablets, show that the starch particles are partially deformed plastically at much smaller pressures than 300 or 400 MPa. That some plastic flow has occurred in the range 100–150 MPa, is shown by the fact that the tablets compressed at above 100 MPa were unbroken and suitable for measurements of dimensions. It has been pointed out that plastic deformation is one of the most important factors for producing firm tablets (David & Augsburger 1977). Thus the K_p-value is not a limit value for plastic deformation although plastic deformation below this pressure cannot be complete. The practical importance of the K_p-value is that it gives an impression of the ease of the plastic deformation and the softness of the material (Heckel 1961b; Kurup & Pilpel 1978). A lower value of K_p tends to favour plastic deformation during compaction. Potato and corn starches are the softest and they are more prone to plastic flow than barley and wheat starches.

From the scanning electron micrographs (cf. Fig. 5) it is evident that most particles of wheat and barley starches are orientated in a certain direction during the compression. The original particles of these starches have a flaky shape (see Fig. 2 and the shape factors F_s in Table 1). Corn and potato starch particles, because of their more spherical shape, do not have any tendency to orientate. According to K_p-values, wheat and especially barley starch are less prone to plastic deformation than corn and potato starch. Thus it seems that higher pressures are needed for barley and wheat starches than for corn and potato starches to obtain the closeness of packing that makes extensive plastic flow possible. The shape of the particles also seems very much to affect the tendency of the starches to deform plastically.

By subtracting the slope of the Heckel plots obtained from the ejected tablet method from the slope obtained from the tablet-in-die-method, and

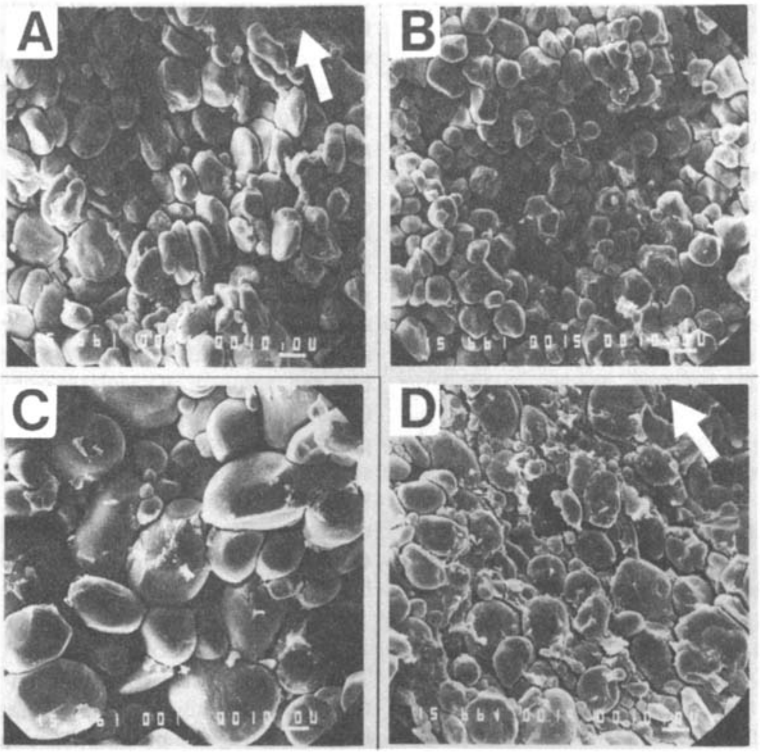

FIG. 5. Scanning electron micrographs of the fracture surfaces of tablets compressed at about 200 MPa. A, Barley starch. B, Corn starch. C, Potato starch. D, Wheat starch. Bar = 10 μm. The orientation direction of particles is shown by the arrow.

taking the reciprocal of that value, it is possible to get a parameter that describes the tendency of a material to recover elastically. This value may be called the yield pressure of elastic deformation, K_e. The values of K_e in MPa are for barley 150, for corn 196, for potato 178 and, for wheat starch 150. Thus wheat and barley starches are most and corn starch is clearly least prone to elastic deformation.

The constants k and Q of the Cooper-Eaton equation are seen in Table 4. The values for k_1 and k_2 in the Cooper-Eaton equation are related to the force necessary for the two stages of compaction (Cooper & Eaton 1962). The values of k_1, the pressure necessary to fill the interparticle void space of the same or greater dimensions than the particles, are lower than the values of k_2, the pressure necessary to fill interparticle void spaces of smaller dimensions than the particles. k_1 and k_2 are small for soft, easily deformable, materials (Cooper & Eaton 1962). These values from equation 2 and also Q-values from equation 3 are small for all the starches studied. This again points to the tendency of the starches to deform.

The values of k_1 of the tablet-in-die-method from equation 2 in the reciprocal pressure range greater than 0·04 MPa^{-1} describe the pressure necessary to fill large voids in the compact. The forces preventing the free packing of different starch particles are so small that they can be overcome by a small compression pressure. The values of k_1 seem to be proportional to the contact area between particles or S_w and d_{sv} (cf. Table 1).

The particles of barley and corn starches easily fill the large voids inside the powder column. The large contact areas between particles of these starches push the free starch particles towards the voids. Thus, the optimum pressure needed to fill large voids, k_1, is small for barley and corn starches but clearly larger for potato and wheat starches, the contact areas of which are smaller as are also the pushing effects. Thus potato and wheat starch particles have to move a relatively longer distance and need larger pressures than corn or barley starches before the larger voids are filled.

The values of k_2 of the tablet-in-die-method from equation 2 in the reciprocal pressure range below 0·02 MPa^{-1}, and Q of the ejected tablet method from equation 3, describe the pressure needed to fill voids smaller than the particles. Because of elastic recovery, the values of Q indicate that higher pressures are needed to fill the smaller voids than are necessary in practice. The values of k_2 and Q should theoretically be proportional to the yield pressure values obtained from the corresponding methods by equation 1. The values of k_2 and K_d describe the tendency of the starches to deform both plastically and elastically and Q and K_p the tendency of the starches to deform only plastically. The tendency of the starches for whole deformation, k_2 obtained with equation 2, seems to be of a similar order to the K_d-values obtained from equation 1. Thus potato and corn starches need a higher pressure for total deformation than barley and wheat starches. The values of Q from equation 3 are greater than the values of k_2 from equation 2 as are also the values of K_p from those of K_d, both from equation 1. Thus, the data from the Cooper-Eaton plots support the findings of the Heckel plots that the starches theoretically need higher pressures for plastic deformation than for total deformation. The order of Q-values for the starches is, however, not the same as that of the K_p-values. The Cooper-Eaton plots do not seem to be as sensitive to elastic recovery of a powder column as the Heckel plots.

The intercept of the two linear parts of the Cooper-Eaton plots of the tablet-in-die-method obtained with equation 2 for the starches in MPa is for barley 42, for corn 45, for potato 44 and, for wheat 42. From the theoretical point of view these values describe the point where the extensive deformation of particles begins and thus they should be proportional to the K_d-values obtained from the Heckel equation. The values of intercepts are all clearly smaller than the values of K_d. The differences between the intercepts are small as are also the differences between the K_d-values. These two parameters show in an equal way that between the starches the total deformation tendency does not indicate great differences. The value of intercept, like the value of K_d, is larger for small particles (corn) than for large particles (potato). The effect of particle size distribution and particle shape are also seen but maybe not as clearly as from the values of K_d. Thus it seems that the intercept of the two stages of Cooper-Eaton plots is good for distinguishing the effects of particle properties on the deformation behaviour of the starches.

CONCLUSIONS

There is no difference between the use of the tablet-in-die-method and the ejected tablet method in determining the packing fractions of the starches using the Heckel equation. However, the first method is better for distinguishing the effect of particle properties on total deformation while the latter is the method of choice especially when plastic deformation is examined. The total fraction of theoretical compaction obtained from the Cooper-Eaton equation is smaller for the ejected tablet method than for the tablet-in-die-method, because of elastic recovery.

Besides yield pressure values, which describe the tendency of a material to undergo total and plastic deformation, it is possible to use the reciprocal of the difference of the Heckel plot slopes obtained from the two methods as a parameter that describes the tendency of a material to deform elastically.

York & Pilpel (1973) and Kurup & Pilpel (1978) have previously concluded that the Heckel equation appears to be more sensitive than the Cooper-Eaton equation in distinguishing the various processes for certain soft pharmaceutical materials. A similar conclusion can be drawn from our results on different starches. However, the intercept of the two stages of Cooper-Eaton plots, and maybe the constant k_2 obtained from the tablet-in-die method, are reasonably good parameters for describing the tendency of a material to deform.

The die filling and rearrangement processes are dependent on the particle size and the size distribution and the shape of the particles. The densification of large starch particles (potato starch) is more the result of die filling and less that of rearrangement. The densification of small particles (corn starch) was the opposite of potato starch. If the starch has a wide particle size distribution (wheat starch) or an irregular particle shape (barley starch) it behaves as if its mean particle size is much smaller than it is in reality.

Plastic flow is that part of deformation which remains after ejection of the tablets. Thus it is a desirable way of deformation, but the existence of profuse elastic deformation is not good for the tableting process. The tendency of the starches to plastic deformation is clearly dependent on the shape of particles. Spherical potato and corn starch particles are more prone to plastic deformation than the more flaky-shaped barley and wheat starch particles. According to the values of yield pressures, corn starch undergoes much plastic flow and only little elastic recovery, and thus is the most favourable among the starches studied. Potato starch also flows plastically with ease. Barley and wheat starches are more elastically deformed than corn and potato starches.

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