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Granulation and compaction of a model system. III. Compaction properties of the granules

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Summary

Granules prepared from glass ballotini by wet massing and screening have been assessed using a double compression technique and calculating work input parameters. Three binders, polyvinylpyrollidone, hydroxypropylmethylcellulose and hydrolysed gelatin, have been used and the resultant granules stored at two relative humidities. Compared to the glass alone, all the binders allowed the compression work to be used more effectively to produce permanent deformation. The effectiveness was dependant on binder type, concentration and moisture content.

Introduction

Previous papers (Cutt et al., 1986, 1987) have described the physical characteristics and stress relaxation properties of granules prepared from a model material, glass ballotini, by massing and screening. Glass ballotini will deform under pressure elastically and by brittle fracture. The influence of binding agents on the compression properties has been interpreted by utilising the double compression technique of de Blaey and Polderman (1970; 1971), and calculating the work involved in compression.

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Materials and Methods

Granules were prepared by wet granulation from lead-glass ballotini (Dragonit 30, Grade 20, Englass, Leicester, U.K.), fractionated to give a mean size of 40 μm (S.D. 8 μm). The granulating agents used were polyvinylpyrrolidone (PVP), average mol. wt. 25,000 Da (Kollidon 25, BASF, Ludwigshafen F.R.G.); hydroxypropylmethylcellulose (HMPC) low viscosity grade (Methocel E15, Colorcon, Orpington, U.K.) and hydrolysed gelatin, average mol. wt. 10–12,000 Da (Byco C, Croda Foods, Widnes, U.K.). Full details of the procedures have been described previously (Cutt et al., 1986). Force–displacement curves suitable for evaluating compression parameters were obtained using an Instron physical testing instrument. Both the upper and lower punch forces were recorded as a function of crosshead displacement during a double compression cycle at a speed of 0.2 cm/min with a 6 s interval between the compression cycles. Corrections were applied to take into account the deflection of the instrument. The areas under the force–displacement curves were quantified by cutting and weighing. The reproducibility of the method was checked by repeated compression of blocks of rubber and was found to be satisfactory. Six replicate experiments were performed at each compression pressure. The weights of glass ballotini and granules used were adjusted to give an initial bulk volume of 0.76 cm^3. A single size fraction of granules, 710–1000 μm, equilibrated at two different relative humidities, was studied. The compression parameters assessed were: gross input (*GI*), elastic deformation (*ED*) frictional work in the first compression (FR_1), net input (*NI*) and the plasticity coefficient (*PC*).

Results and Discussion

The calculated parameters used to describe the compression of the glass beads are shown in Fig. 1. The total work performed on the ballotini by the upper punch during the first compression, the gross input (*GI*), was shown to increase almost linearly with an increase in compression pressure. This gross input comprised of: (a) the work required to rearrange the particles and increase their packing density; (b) the work required to overcome die wall friction; (c) the work required for elastic and plastic deformation; (d) the work required to cause fracture; and (e) the work used in the formation of interparticulate bonds.

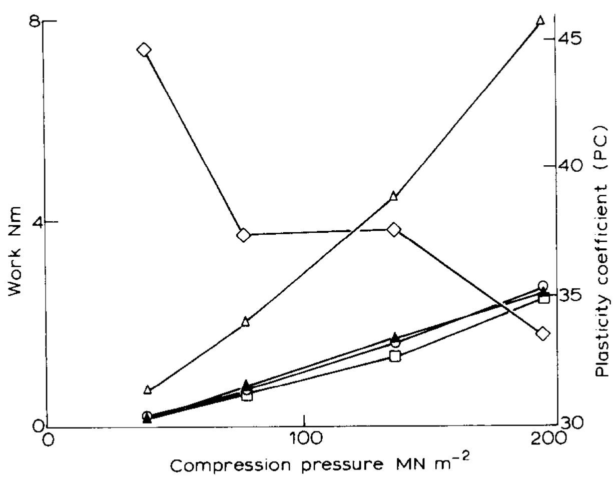

Fig. 1. The work involved in the compression of glass ballotini. △, *GI*: ○, *ED*; □, FR_1; ▲, *NI*; ◇, *PC*.

The elastic deformation (*ED*) of the glass beads was estimated from the work expended during the second compression cycle. This was also shown to increase linearly over the range of compression pressure used, as was the work used in friction (in the first compression, FR_1). The net input (*NI*), or work used in permanent deformation of the glass beads, was also shown to be a linear function of the compression pressure. This represented a progressive fracture and subsequent increase in surface area of the glass beads with increasing pressure, as the ballotini permanently deformed only by brittle fracture, and not by plastic deformation. The fraction of the total work not used to overcome elasticity, represented by the plasticity coefficient (*PC*) was calculated from the ratio, $(NI/GI) \times 100\%$. The decrease in value of this coefficient with increasing compression pressure is shown in Fig. 1. This indicates that as the compression pressure was increased, the proportion of total work used for permanent deformation decreased. At higher pressures, greater proportions of the work used in compression were necessary to overcome friction and elasticity. The actual values of the plasticity coefficient show that the percentage of the total compression work used for permanent deformation, in this case the fracture of the glass beads, was less than 50%, over the range of pressures studied. Comparable proportions of the gross input were used for elastic deformation of the glass beads, indicating that the bed of glass beads was considerably elastic in nature. Therefore, on removal of the compressive load, a significant amount of elastic expansion occurred at all 4 pressures studied.

The effect of granulation of glass beads on the work involved in their compression is shown in Table 1. The results for the glass beads are given in Table 2 for comparison. The following general conclusions can be drawn. At the two lower pressures, generally more work was utilized to permanently deform the granules than the glass (as

TABLE 1

The work involved in the compression of glass granules containing 3% w/w binder

S.E.R.H. = storage equilibrium relative humidity.

Granules (weight, g)	S.E.R.H.	Compression pressure ($MN \cdot m^{-2}$)	*GI* (Nm)	*ED* (Nm)	FR_1 (Nm)	*NI* (Nm)	*PC*
3% PVP (0.5853)		39	1.261	0.150	0.235	0.874	69.31
		77	2.137	0.435	0.389	1.374	64.30
		136	3.498	0.813	0.759	1.939	55.43
		194	5.243	1.546	1.226	2.522	48.10
3% Byco (0.4573)	12%	39	1.306	0.145	0.149	1.010	77.34
		77	2.045	0.362	0.344	1.403	68.61
		136	3.300	0.785	0.666	1.860	55.36
		194	4.856	1.110	1.382	2.417	49.77
3% HPMC (0.5312)		39	0.871	0.159	0.184	0.519	59.59
		77	1.648	0.422	0.514	0.774	46.97
		136	2.592	0.765	0.680	1.211	46.72
		194	4.456	1.306	1.761	1.442	32.36
3% PVP (0.5853)		39	0.613	0.147	0.128	0.336	54.81
		77	1.005	0.370	0.230	0.470	46.77
		136	1.960	0.819	0.395	0.759	38.72
		194	3.396	1.580	0.604	1.286	37.87
3% Byco (0.4573)	65%	39	1.215	0.109	0.150	0.903	74.32
		77	1.885	0.352	0.386	1.212	64.30
		136	2.943	0.774	0.549	1.632	55.45
		194	4.008	1.160	1.177	1.722	42.96
3% HPMC (0.5312)		39	0781	0.139	0.174	0.444	56.85
		77	1.318	0.422	0.276	0.687	52.12
		136	2.522	0.835	0.650	1.058	41.95
		194	3.816	1.006	1.493	1.375	36.03

shown by the *NI* values), the additional work being utilized in the granule deformation and fracture processes which were apparent at these lower pressures. The *ED* and FR_1 values were also generally lower for the granules than the glass, irrespective of the compression pressure, indicating that the binders were effective in reducing both the reversible elastic deformation, and the work loss to friction during compression. These lower values were responsible not only for the lower work needed to compress the granules at the two higher pressures (compared with the glass

TABLE 2

The work involved in the compression of glass ballotini

Compression pressure ($MN \cdot m^{-2}$)	*GI* (NM)	*ED* (NM)	FR_1 (NM)	*NI* (NM)	*PC*
39	0.711	0.183	0.198	0.318	44.73
77	2.088	0.722	0.655	0.782	37.45
136	4.430	1.622	1.368	1.667	37.63
194	7.746	2.696	2.502	2.601	33.57

alone), but also for the greater deformation efficiency of the compression work in the granules (as represented by the *PC* value). The total amount of work needed to compress dry granules and the work used in their plastic deformation, fracture and binding (*GI* and *NI*) were higher than for the corresponding damper granules. Although the damper granules generally had lower *PC* values, this indicated only that less of the total work input was necessary for their irreversible deformation.

The total work of compression (*GI*) used to compact the granules after storage at 12% R.H. was highest for the PVP granules, and after storage at 65% R.H., for the Byco granules. Of the granules stored at 12% R.H., those containing HPMC as the binder required the least compression work, due to the significantly weaker structure of these granules (Cutt et al., 1986). However, after storage at 65% R.H., the PVP granules required the least work for their consolidation. The elastic deformation of the PVP granules was the greatest after equilibration at 12% R.H., whilst that of the Byco granules, the lowest. Of the granules stored at 65% R.H., the rank order for work necessary to elastically deform the compacts was: PVP > Byco > HPMC at the highest pressure. The work expended to overcome friction during compression for the granules stored at 12% R.H. was inconsistent for binder type, and emphasised that this parameter was the least accurately determined by this double compression technique. For the granules stored at 65% R.H., the rank order for friction was generally: HPMC > Byco > PVP, and was dependent on the actual granule moisture contents, which were a function of the hygroscopicity of each binder. The *NI* values showed that the compression work used for permanent deformation of the PVP and Byco granules after storage at 12% R.H. was similar, but significantly greater than that used to compress HPMC granules. However, after storage at 65% R.H., significantly less work was necessary to compact the PVP granules than the Byco granules, those containing HPMC exhibiting intermediate values. The ease of compression of these damp PVP granules is attributed to the fact that PVP absorbs sufficient moisture to become semi-solid at 65% R.H. Hence these granules offered little resistance to compression. The plasticity coefficient (*PC*) showed the rank order: Byco > PVP > HPMC for granules stored at 12% R.H., and Byco > HPMC > PVP at 65% R.H., paralleling the *NI* values found for these granules. Hence Byco was found to form strong granules which required more work to compress them than granules of the other two binders, a greater proportion of this compression work being used to permanently deform the Byco granules, either by rearrangement, plastic deformation or crushing.

Granules were also prepared from hydrophobic glass beads (Mohammad and Fell, 1982; Cutt et al., 1986) using PVP, as a binder. Both the total work used in compression (*GI*) and the work used for irreversible deformation (*NI*) was found to be lower for these granules than corresponding granules prepared from hydrophilic glass. This can be attributed to the weakness of the granules formed from the hydrophobic glass (Cutt et al., 1986).

The results presented show that in the presence of a binding agent, the compression work was used more effectively to produce permanent deformation. The reduction in elastic deformation work, as a result of granulation, shows that binders may be useful in reducing capping problems exhibited by such materials as paracetamol, which, like the glass beads, exhibit much elastic, but little plastic deformation. Although all 3 binders increased the ease of deformation of the model particulate system, and in particular the plastic deformation during compression, the amount by which they did so was dependent on both the concentration of binder and the moisture content of the granules, as well as the binder type.

In the optimization of compression with respect to binder type, granule size and moisture content, the selection of the optimum level of each variable would be based on seeking those which satisfy any required specifications and also produce the following: a low gross work input; a high plasticity coefficient (and therefore net work input); low friction work; and low elastic deformation. Following this selection, the optimal granules should be easily compressible, with the most efficient conversion of compression work to plastic deformation, fracture and bonding within the compressed material. Friction during compression would be low, reducing wear on the compression tooling, and the tendency for capping would be minimized by the low elasticity of these granules.

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