THE STRENGTH OF COMPRESSED TABLETS

Part II. The Bonding of Granules during Compression

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The deformation of tablet granules has been followed over a wide pressure range by preparing and compressing sucrose granules with coloured surfaces. By examination of the fracture of the compacts in a strength test, it has been possible to determine the structural role of the granule at these pressures.

In investigating the factors affecting the strength of a compressed tablet, it was considered desirable to construct a qualitative picture of the behaviour of tablet granules during compression and subsequent fracture.

Seelig and Wulff[1] divide the mechanism of the compression of powders into three stages. (*a*) Packing; during this stage, interparticular friction absorbs most of the applied energy. (*b*) Elastic and plastic deformation; the applied energy is mainly consumed as interparticular and die-wall friction. (*c*) Cold working, with or without fragmentation.

Although these hypotheses were devised to explain the compression of powdered metals, Seth[2] used them as an empirical account of the formation of pharmaceutical tablets, considering that fragmentation caused the initial increase in the total surface of the tablet reported by Higuchi, Rao, Busse and Swintosky[3]. This communication is a qualitative discussion of these factors.

A sharp demarcation of the granule boundary was obtained by Strickland, Nelson, Busse and Higuchi[4]. Granules were covered with activated charcoal in a successful attempt to determine the distribution of lubricant in compressed tablets.

In our work, observation of the boundary was made possible by colouring the outer layer of the granule.

Experimental

A roughly spherical sucrose granulation was prepared in a rotating coating pan. A small quantity of sucrose solution was added to crystal nuclei and the mixture dried. This process was repeated until an approximately 14–22 mesh material was obtained. This size was chosen because of its suitability for subsequent examination in the tablet matrix. The final addition of syrup contained a vivid dye, so that each granule received a thin coating of colour. Six batches, each of a different colour, were prepared and screened.

After determination of its density, the granulation was compressed on the instrumented tablet machine already described[5], and the compression forces recorded. Tablets were produced at five pressure levels with eight tablets in each series. A constant weight of fill was used, calculated to give a tablet 0·4 cm. thick at zero porosity. As sucrose exhibits high frictional effects when compressed, the punch and die surfaces were lubricated with a solution of stearic acid in acetone and carbon tetrachloride. After measurement of weight and thickness, six tablets from each series were subjected to the crushing test previously described[5]. The two remaining tablets were used for examination by microscope. An upper surface and a fractured surface of a tablet from each series was photographed. Examples from two series are given in Figure 1.

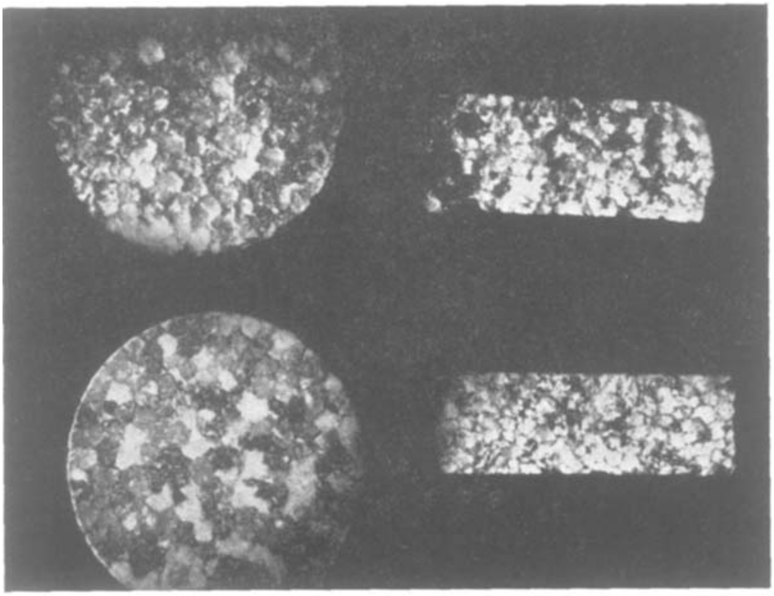

FIG. 1. Examples of tablet surfaces and fracture. The upper tablets were made at a mean compaction pressure of 530 kg./sq. cm. and the lower at 2,020 kg./sq. cm.

RESULTS

The values of mean compaction pressure, P_m, crushing force and porosity are given in Table I.

The relationship between porosity and the resistance to crushing is given in Figure 2. The results are plotted according to the equation:

$$F_c = F_{co}e^{-bp}$$

where F_c is the crushing force, F_{co} the crushing force at zero porosity, p is the porosity and b is a constant. This equation was empirically derived by Ryshkewitch and Duckworth[6] for porous ceramic materials and has been found applicable to the compaction of some drugs.

At low pressure (530 kg./sq. cm.), examination of the upper surface still reveals the original spherical configuration of the granules and interstices. With increase in pressure, the granule was seen to be distorted massively, accommodating the changes in neighbouring granules, and producing a close interlocking network, with the elimination of the surface interstices. The surface of the granules shows slight white striations at low pressure indicating a small amount of surface cleavage which reveals the uncoloured material of the bulk of the granule. This effect increases progressively with increase of pressure until the surface layer was seen as coloured fragments on a white background.

TABLE I

MEAN COMPACTION PRESSURE, CRUSHING FORCE AND POROSITY

Mean compaction pressure P_m kg./sq. cm.	Crushing force F_c kg.	Porosity p per cent
2,020	10·13	7·55
1,655	7·47	9·29
1,375	5·82	10·86
855	2·63	16·25
530	1·58	19·92

Mean values are quoted

Examination of the fractured surface also gave the opportunity to follow the interlocking progression but in a plane at right angles to the surface and parallel to the direction of the applied force.

The actual line of fracture in the strength test is of great importance. In the low pressure series, 530 kg./sq. cm., the line of fracture usually

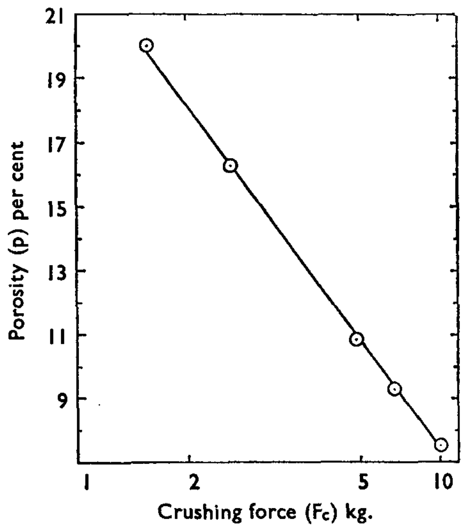

FIG. 2. The relation between porosity and crushing force.

follows the boundary of the granules, and leaves a coloured surface for observation. At a pressure of 855 kg./sq. cm., the proportion of granule surface visible on the fractured surface is much smaller and the tendency to break across the granule predominates. At 1,375 kg./sq. cm. and above, this is the only mode of failure.

DISCUSSION

The stages of powder compression listed in the introduction will not be clearly defined but will overlap to an extent varying with the observed material. The relation between the porosity and the crushing force is found to be linear over the observed range, suggesting that there is no sharp transition from one mode of behaviour to another. Also, it may be concluded that the decrease in voidage due to packing is largely complete in the tablets produced at even the lowest pressures. These tablets exhibit a porosity of under 20 per cent whereas the minimum porosity without deformation for a material of this size range and shape is over 25 per cent.

Examination of the fractured surfaces indicates the pressure range over which the structural identity of the granule is lost due to fragmentation. Before this, failure occurs mainly around the granule. This is shown in the series at 530 and to a lesser extent at 855 kg./sq. cm. where the strength of the intergranular bond is increasing. Fracture across the granule indicates that the bond between adjacent particles of different granules is at least as strong as the bonding forces in the granule. Over this range, therefore, there must have been considerable fragmentation and rebonding of the fresh surfaces as suggested by Higuchi's report[3] of an increase in the total surface area, followed by a decrease, during compression. Where the fractured surface shows that cleavage has taken place independently of the original granule configuration, fragmentation and rebonding has destroyed the original structural units. In view of this, it is doubtful if the apparent interlocking in which each granule participates on compression has the structural significance found in the compression of some of the dendritic materials used in powder metallurgy and suggested as a mechanism in the bonding of tablets by Seth.

The minimum porosity obtained was 7·55 per cent although no voids are apparent in a microscopic examination. This also, is suggestive of a process of fragmentation and rebonding during which the void spaces found in the loose aggregate of granules (Series 1 and 2) are redistributed as a fine network (Series 3, 4 and 5).

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After Mr. Ganderton presented the papers there was a DISCUSSION. The following points arose.

The work had not yet included the use of porous granules because initially it was intended to keep the system simple; the work should be continued on a rotary machine because of the different mode of compression.