A preparative technique for studying the structure of compacts

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The prediction of three-dimensional configuration from analysis of a two-dimensional section would be useful in determining the structure of a compact. Relations can be predicted by stereology in the presence of random distributions. In a preliminary attempt to apply this technique to pharmaceutical systems it was necessary to prepare two-dimensional sections and test the systems for randomness.

The method employed was that described by Smith (1972). Compacts were prepared of sodium chloride, copper powder, magnesium carbonate, limestone and potassium permanganate using a hydraulic press. The compacts were sectioned using a microtome and embedded in resin, or first embedded in resin and cut by a band-saw. Random dispersions of particles in resin were prepared by simple mixture. In preliminary experiments, an epoxy resin casting system was used, but later this was replaced by a styrene polyester resin owing to partial dissolution of one of the materials investigated (sodium chloride) by the epoxy resin.

The specimens were examined microscopically and photomicrographs were obtained using the light microscope and also the scanning electron microscope. Some preliminary examination of the samples was carried out by the Quantimet 720.

In order to obtain resin penetration of the compacts the original material had to be of suitable particle size (e.g. for sodium chloride, 420–600 μm) and also a low pressure of compaction (about 1 MN m^{-2}) was desirable. High porosity by itself did not ensure resin penetration as was found with limestone (36%) and magnesium carbonate (39%).

Satisfactory examination by the Quantimet demanded good contrast between particles and resin because the instrument is intended for black and white specimens only. Black, white and red pigments were used in the resin casting systems to improve the contrast. To overcome the difficulty caused by the transparency of sodium chloride crystals, an etching technique was used, the surface layers of crystals being dissolved by water and the cavities filled by a thick paste of plaster of Paris.

The photographic evidence obtained shows that this technique of embedment can be applied to compacts and dispersions under certain conditions. Provided randomness in these systems can be established it should be possible to predict various features of compacts by means of stereological relation.

REFERENCE

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Indentation hardness testing of tablets

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A simple Brinell hardness tester has been used to measure hardness and elasticity over the faces of tablets prepared from direct compression bases and other materials.

The tester incorporates a displacement transducer to measure the depth of travel of a 1·59 mm sapphire sphere into a tablet under a constant load of 400 g, and also the relaxation of this identation on removal of the load. 13 mm tablets were prepared from 500 mg of powder (with 1% magnesium stearate) at a pressure of 50 MN m^{-2} and the variation in the indentation hardness and elastic recovery was found by making 20 indentations in each of 5 concentric zones on both faces (Ridgway, Aulton & Rosser, 1970).

For each of the tablets prepared from the direct compression bases, the variation over their surfaces was small, $\pm$4% over the inner four zones. The mean hardness of the outer zone was 15% lower than that of the inner zones. This uniformity suggests even distribution, and hence efficient transmission, of forces during the manufacture of the tablet; this is indicative of a good tableting material. This is emphasised by the greater variations

observed on the other materials (up to 30% overall). The mean Brinell hardness for each tablet is summarized in Table 1, which shows that the hardness of the direct compression base tablets falls in the range 30 to 50 MN m^{-2}. The test can also show the effects of formulation changes, as indicated by the increase in hardness of paracetamol tablets prepare from powder, a granulation (with PVP and 8% starch) and a direct compression form respectively. Similarly, Asagran produces harder tablets than aspirin crystals.

Table 1

Brinell hardness	MN m^{-2}
Sucrose	61·2
Emcompress	50·0
Sta-Rx 1500	49·0
Paracetamol DC	35·7
Asagran	34·4
Avicel PH 101	32·8
Celutab	30·8
Aspirin gran. crys.	27·6
Paracetamol grans.	27·1
Lactose anhyd.	18·6
Paracetamol powder	12·3

Table 2

Height of relaxation (μm) / Indentn under load (μm)	Elastic Quotient
Avicel PH 101	0·61
Paracetamol grans.	0·60
Paracetamol DC	0·53
Lactose anhyd.	0·53
Paracetamol powder	0·51
Celutab	0·50
Sucrose	0·43
Emcompress	0·41
Sta-Rx 1500	0·39
Asagran	0·36
Aspirin gran. crys.	0·35

It is suggested that tableting materials which deform plastically with little elastic recovery should produce better quality tablets than more resilient materials. Thus, an elastic quotient has been calculated; this is the fraction of the indentation which rebounds elastically on removal of the load. The quotients are listed in Table 2 which indicates that the tablets prepared from the aspirin and direct compression bases generally have a lower elastic recovery than the poorer tableting materials e.g. paracetamol. An exception is Avicel which shows a high elastic recovery. This may be explained by its hollow microfibrillar structure (Marshall, Sixsmith & Stanley-Wood, 1972).

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The characterization of the mechanical strength of tablets

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Although it has been shown by Fell & Newton (1970) that the "scatter" in the fracture strength of nominally identical tablets can be reduced by ensuring that the specimens fail in tension, variations in strength will be observed, no matter how carefully controlled the test conditions. This variability is an inherent feature of a material which cannot deform plastically under increasing stress (i.e. a *brittle* material) and, because of it, a statistical treatment is essential for a full definition of the mechanical strength of such materials.

The Weibull distribution (Weibull, 1951) offers a valid mathematical model of this particular form of variability (Davies, 1971) and is being increasingly used for material characterisation and component failure predictions in the case of high-strength ceramics (Stanley, Sivill & Fessler, 1974). The two principal assumption are (i) that the material is isotropic and contains a statistically uniform distribution of flaws and (ii) that once a crack has initiated from a flow it will propagate without further increase in load, resulting in fracture. The