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The effect of sinusoidal vibration on the uniformity of packing of powder beds

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Sinusoidal vibration, at a number of defined sets of conditions, has been applied to packings of certain particle size fractions of lactose. The distribution of local porosity within these vibrated packings was determined using a gamma-ray attenuation technique, and could be compared with porosity data for samples not subjected to vibration. It was found that the application of vibration in a vertical mode markedly increased the uniformity of packing; horizontal vibration was less effective in this respect. The relationship between local porosity and position with a packing, observed in most non-vibrated samples, was generally absent from vibrated packings.

In many powder handling processes, porosity or bulk density is an important quantity, and there are instances in which uniformity of packing is also desirable. For example, the weight variation (and consequently the dose variation) of tablets and dosator-filled hard gelatin capsules, can be significantly influenced by local porosity variations in the bulk powder or granule feed.

The overall porosity of a powder packing is dependent on the physical properties of the particles, such as size, shape and surface characteristics. The level of porosity can also be influenced by the method of preparation of the powder bed. Kolbuszewski (1950), Macrae & Gray (1961), and Eastwood et al (1969) found that the packing density of free-flowing materials was affected by the velocity and intensity of their deposition, minimum porosity generally being achieved at low intensity and high velocity. Woodhead et al (1983) studied the effects of sinusoidal vibration on the porosity of powder beds, and found that porosity could be minimized by selection of optimum vibration conditions.

Similarly, the local variability of porosity within powder packings has been shown to be influenced by many of the above factors. For example, in a study of the effect of the method used in powder deposition on the resulting packing uniformity of cylindrical beds, Woodhead & Newton (1983) found some methods consistently produced more uniform packings than others. In particular, low variability in local porosity could be achieved by even distribution of particles over the whole cross-section of the growing bed. Van Brakel & Heertjes (1974) studied the influence of various deposition and vibration conditions on uniformity of packing, and attained optimum homogeneity by depositing powder at a specific velocity and intensity into containers being horizontally vibrated.

Measurement of local porosities within powder beds is best made with non-destructive techniques. Woodhead et al (1982) described a gamma-ray attenuation method whereby the distribution of porosity within cylindrical packings of lactose could be determined. The principle of the technique is that when a narrow beam of gamma radiation passes through a powder packing of thickness L, the measurable count rate is reduced from N_o to N, according to the following equation:

$$N = N_o \exp\{-\mu(1-\varepsilon)L\} \quad (1)$$

The linear attenuation coefficient, μ, of the powder material at the relevant photon energy can be determined by experiment. Alternatively, It may be estimated from tabulated data (Hubbell 1969). It is then possible to determine, from equation 1, the local porosity, ε, of that part of the packing traversed by the radiation beam.

Radioactive decay is a random process, and therefore the variability of a measured count rate is described by the Poisson distribution. As a result, reproducibility is improved by counting for longer time intervals. To measure a local porosity value, ε, with confidence intervals of $\pm\Delta\varepsilon$, at a probability level denoted by p, the counting time interval must be sufficiently long for the reference ('blank') count to exceed a certain minimum value, $N_{o_{min}}$. This can be calculated from equation (2), the derivation of

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which appears elsewhere (Kurz 1972; Woodhead et al 1982).

$$\frac{N_{o_{min}}}{p^2} = \left[\frac{\exp\{-\frac{1}{2}\mu(1-\varepsilon-\Delta\varepsilon)L\}+\exp\{-\frac{1}{2}\mu(1-\varepsilon+\Delta\varepsilon)L\}}{\exp\{-\mu(1-\varepsilon-\Delta\varepsilon)L\}-\exp\{-\mu(1-\varepsilon+\Delta\varepsilon)L\}}\right]^2 \quad (2)$$

In this work, the gamma ray attenuation technique is used to detect local porosity variations in packings of lactose subjected to various vibration conditions. Comparative porosity data for non-vibrated packings are also presented.

MATERIALS AND METHODS

Crystalline α-lactose monohydrate was used as a model powder system. Three particle size fractions were selected: +18·7–26·5, +37·5–53 and +75–105 μm. These materials had been shown to have similar particle shapes and angles of internal friction (Woodhead et al 1983).

All packings were prepared in identical cylindrical aluminium containers, of internal diameter 19 mm and depth 44 mm. Vibration was applied by mounting a cylinder on an electromagnetic vibrator, driven by an oscillator/amplifier (Models VP3 and TA 120 respectively, Derritron Electronics Ltd, Hastings, Sussex). A piezo-electric accelerometer (D Birchall Ltd, Mildenhall, Suffolk), also mounted on the vibrator, was connected to a Portable Vibration Analyser (PVA, Derritron), enabling the frequency, acceleration and amplitude of the applied vibration to be monitored.

Local porosity measurements were made using the gamma-ray attenuation system described by Woodhead et al (1982). A 200 mCi (7·4 GBq) sealed, directional point source of Americium-241 transmitted a collimated cylindrical beam of 60 keV gamma radiation vertically through a prepared powder bed. Those photons not absorbed or scattered by the powder were detected by a sodium iodide crystal and recorded by a scaler-ratemeter (Ortec Ltd, Luton, Beds). Accurate positioning of the powder sample was ensured by means of a micromanipulator (Model 932/T, Prior, Bishops Stortford, Herts).

Methods

Each packing to be vibrated was prepared by depositing powder into an aluminium cylinder to which was attached an extension collar that increased its depth by 28 mm. Powder in excess of the amount required to fill this was deposited and then the upper surface of the packing was levelled using a straight edge. Initial porosity was calculated from the weight and bulk volume of the packing, and the apparent particle density of lactose, determined by air pycnometer to be 1·55 g cm^{-3}. The cylinder was then attached, upright, to the vibrator table, and a well-fitting cylinder of nylon 4 cm long that closely fitted the extension collar was positioned on top of the packing to maintain a level surface during vibration. After vibration, the nylon cylinder and collar were removed, and excess powder was scraped off using a straight edge. The final porosity was then calculated.

Three deposition methods were employed, these having been found to give relatively uniform packing of non-vibrated samples in earlier work (Woodhead & Newton 1983). Briefly, they comprised (i) pouring directly from a jar, (ii) deposition from a vertical tube through a circular orifice, possible only for the +37·5–53 μm fraction (12 mm diameter orifice) and the +75–105 μm fraction (4 mm diameter orifice), (iii) deposition from a vibrating chute into a container moving repeatedly through the falling stream of powder, thus building each packing in even layers.

For each particle size fraction, previous experiments had established optimum vibration conditions for consolidation (Woodhead et al 1983). As a result, emphasis was placed on vertical vibration at 6*g* acceleration and certain optimum frequency settings, though other conditions such as lower acceleration and horizontal vibration were also investigated.

Packings were prepared in groups of four, and local porosity values were determined by gamma-ray attenuation measurements. The time interval for gamma counting, chosen to ensure a specific degree of confidence in each porosity value, was calculated from equation (2), into which the following values were substituted: $p = 1{\cdot}96$ (corresponding to 95% confidence at ∞ degrees of freedom), $\mu = 0{\cdot}288$ cm^{-1} (calculated from tables, Hubbell 1969), $\Delta\varepsilon = 0{\cdot}005$. For each packing, the values of L and ε corresponded to the measured bed depth and overall porosity respectively. Each sample was placed in the gamma ray beam, and 'counts' of the calculated duration were recorded in each of 19 positions forming a regular matrix over the cross-sectional area of the packing. These positions comprised the centre, plus six each at radial distances of 4, 7 and 8 mm. Reference ('blank') counts were recorded periodically with an identical empty container in the radiation beam.

Local porosity values were calculated from equa-

tion (1) using an expression for the attenuation coefficient of lactose that was established by Woodhead et al (1983).

This expression

$$\mu = 0{\cdot}289 + 0{\cdot}0215\varepsilon \qquad (3)$$

indicated that the attenuation coefficient is a function of sample porosity, hence an iterative process was employed to calculate local porosity from equations (1) and (3).

The variability in local porosity was characterized using a statistical package (Genstat V), consisting largely of a variance analysis of each group of four replicates of the 19 positions of measurement. The within-samples variance ratio indicated any significant relationship between local porosity and radial position, and this relationship was tested for linear fit. The between-samples variability was also represented by the appropriate variance ratio. The grand mean of all 76 values provided a measure of the overall level of porosity, whilst minimum and maximum values enabled the spread of local porosity existing within a group of packings to be calculated. The total sum of squares of deviations from the grand mean gave a comparative measure of overall variability. Mean porosity values were also calculated for each radial distance in a group of four packings.

RESULTS AND DISCUSSION

Porosity data for vibrated and non-vibrated packings of lactose particle size fractions are presented in Tables 1–3, in which the term 'r' denotes radial distance from the centre of each packing. The significance level of the variance ratios is indicated by asterisks, where * denotes significance at the 95% probability level and ** denotes significance at the 99% level (Fisher & Yates 1970).

For each particle size fraction, the overall porosity of non-vibrated samples, as reflected by the grand mean local porosity, was dependent on deposition method, but in all cases, porosity was reduced by the application of vibration, as expected. The +18·7–26·5 μm size fraction, being cohesive, produced packings of highest porosity before vibration, but showed the greatest degree of consolidation under given vibration conditions. The +37·5–53 μm size fraction, however, produced beds of lowest overall porosity, closely followed by the +75–105 μm fraction. Such porosities, around 0·39, are probably near the limit of consolidation for these systems; any further densification would involve deformation and fragmentation of individual particles.

For all three size fractions, vertical vibration, at a suitable frequency and an acceleration of 6*g*, proved to be much more effective than horizontal vibration in producing consolidation. Indeed, in one instance, vertical vibration at 2*g* resulted in a lower porosity than did horizontal vibration of 6*g* (+18·7–26·5 μm fraction; deposition method (i)).

Vertical vibration led to a decrease in the variability of local porosity as reflected by 'total sum of squares' and local porosity spread, when compared with the corresponding data for non-vibrated samples. Only one exception to this trend was observed, i.e. the vibration of the +18·7–26·5 μm size fraction at a frequency of 150 Hz. It could be argued that 150 Hz is not the optimum vibration frequency, being less effective than 100 Hz in reducing mean

Table 1. Statistical analysis of local porosity data for lactose, particle size +18·7–26·5 μm.

Filling method	(i)	(i)	(i)	(i)	(i)	(iii)	(iii)	(iii)
Vibration conditions:								
Frequency (Hz)	—	100	100	150	500	—	100	500
Acceleration (*g*)	—	2	6	6	6	—	6	6
Direction†	—	V	V	V	H	—	V	H
Grand mean local porosity	0·670	0·563	0·429	0·473	0·579	0·625	0·440	0·536
Local porosity spread	0·049	0·025	0·043	0·064	0·069	0·050	0·037	0·030
Mean local porosity r = 0 mm	0·662	0·560	0·421	0·472	0·591	0·616	0·437	0·532
r = 4 mm	0·665	0·564	0·427	0·476	0·586	0·621	0·441	0·536
r = 7 mm	0·671	0·562	0·430	0·473	0·575	0·624	0·440	0·538
r = 8 mm	0·675	0·563	0·431	0·471	0·575	0·631	0·440	0·538
Variance ratios								
Within samples	4·9**	2·7	6·0**	4·6**	11·9**	9·7**	0·7	0·96
Linear fit	14·2**	0·3	12·9**	9·3**	26·3**	17·5**	0·05	1·3
Between samples	4·2**	40·2**	36·9**	89·9**	38·5**	8·9**	37·0**	3·5*
Total sum of squares × 10^4	95	27	52	241	148	62	46	34

† H = Horizontal, V = vertical.
* Significant at the 95% level of probability. ** Significant at the 99% level of probability.

Table 2. Statistical analysis of local porosity data for lactose, particle size +37·5–53 μm.

Filling method	(i)	(i)	(i)	(i)	(i)	(ii)	(ii)	(ii)	(iii)	(iii)	(iii)
Vibration conditions:											
Frequency (Hz)	—	200	200	100	100	—	100	100	—	100	100
Acceleration (*g*)	—	2	6	6	6	—	6	6	—	6	6
Direction†	—	V	V	V	H	—	V	H	—	V	H
Grand mean local porosity	0·565	0·504	0·405	0·389	0·467	0·526	0·394	0·453	0·499	0·395	0·418
Local porosity spread	0·056	0·024	0·024	0·025	0·058	0·041	0·020	0·039	0·041	0·021	0·035
Mean r = 0 mm	0·555	0·499	0·406	0·388	0·467	0·511	0·396	0·453	0·497	0·395	0·418
local r = 4 mm	0·562	0·502	0·406	0·391	0·470	0·518	0·395	0·453	0·501	0·396	0·421
porosity r = 7 mm	0·567	0·505	0·404	0·388	0·467	0·529	0·392	0·451	0·495	0·394	0·417
r = 8 mm	0·568	0·505	0·404	0·389	0·466	0·535	0·394	0·455	0·501	0·395	0·415
Variance ratios											
Within samples	1·5	5·2**	1·6	3·2*	0·4	39·2**	3·6*	2·8*	2·8	0·9	21·3**
Linear fit	3·3	7·3**	4·0*	3·4	0·9	110**	2·6	2·2	0·3	0·6	53·1**
Between samples	2·8	9·7**	14·7**	12·4**	31·2**	6·0**	2·8*	99·4**	1·4	10·5**	41·4**
Total sum of squares × 10^4	172	15	18	20	230	79	13	75	59	17	44

† H = Horizontal, V = vertical.
* Significant at the 95% level of probability. ** Significant at the 99% level of probability.

Table 3. Statistical analysis of local porosity data for lactose, particle size +75–105 μm.

Filling method	(i)	(i)	(i)	(i)	(ii)	(ii)	(ii)	(iii)	(iii)	(iii)
Vibration conditions:										
Frequency (Hz)	—	150	150	200	—	150	120	—	150	120
Acceleration (*g*)	—	2	6	6	—	6	6	—	6	6
Direction†	—	V	V	V	—	V	H	—	V	H
Grand mean local porosity	0·523	0·508	0·407	0·418	0·530	0·402	0·429	0·446	0·402	0·411
Local porosity spread	0·043	0·034	0·031	0·035	0·036	0·025	0·025	0·054	0·023	0·053
Mean local r = 0 mm	0·516	0·507	0·408	0·418	0·514	0·401	0·434	0·434	0·404	0·403
porosity r = 4 mm	0·518	0·508	0·408	0·421	0·527	0·402	0·430	0·439	0·404	0·406
r = 7 mm	0·524	0·508	0·406	0·417	0·534	0·402	0·425	0·444	0·401	0·409
r = 8 mm	0·526	0·508	0·407	0·415	0·533	0·401	0·433	0·457	0·401	0·421
Variance ratios										
Within samples	4·0*	0·1	2·9*	6·3**	30·6**	0·6	13·6**	16·1**	4·6**	18·7**
Linear fit	10·1**	0·1	3·7	15·3**	49·5**	0·9	5·4	47·3**	9·3**	52·6**
Between samples	5·0**	97·5**	156**	69·7**	2·6	43·8**	0·6*	1·0	89·9**	1·1
Total sum of squares × 10^4	75	52	47	56	36	20	24	116	31	76

† H = Horizontal, V = vertical.
* Significant at the 95% level of probability. ** Significant at the 99% level of probability.

porosity. Horizontal vibration was much less consistent in that the variability in local porosity fell in some cases and rose in others.

In non-vibrated packings, there was generally a significant relationship between local porosity and radial position, central areas being of lower porosity. This is not thought to be due to a direct wall effect as discussed by Ridgway & Tarbuck (1966), which normally influences packing over a distance of only a few particle diameters from the container wall. It is more likely to be due to the mechanism of particle deposition. Failure to avoid preferential deposition towards the centre of the container will result in particles rolling outward towards the wall, effectively depositing at a relatively low velocity. This will lead to a higher porosity (Kolbuszewski 1950; Macrae & Gray 1961).

Examination of mean local porosity values at each radial distance for vibrated samples, together with the corresponding variance ratios, revealed that in only two out of 21 instances was there a clear increase in local porosity with increasing distance from the centre of the packing. There was a general tendency for variability of local porosity to fall largely between samples instead. This is reflected by the very high between-samples variance ratios, as

shown in Tables 1–3. This suggests that vibration, even when applied in a controlled manner, gives rise to poor reproducibility of overall porosity between otherwise identical samples. This is not the case. Previous experiments (Woodhead et al 1983), showed that vertical vibration at selected conditions resulted in final porosities with coefficients of variation typically around 1%. The between-samples variance ratios quoted here can be misleading if not considered in the light of the total variability in local porosity, which, for vibrated packings, is normally very low.

In conclusion, these experiments have demonstrated that the application of sinusoidal vibration to powder packings not only decreases the overall porosity of each packing, but in most cases, the variability of local porosity is also reduced.

This is especially true for vertical vibration, which is significantly more effective than horizontal vibration in reducing both the overall level and the variability of porosity. After vibration, the statistical variance of local porosity values falls largely between, rather than within samples, indicating a high degree of uniformity of packing within each sample. The relationship between porosity and radial position, observed in many non-vibrated packings, is generally absent from vibrated packings.

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