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The vibratory consolidation of particle size fractions of powders

P. J. WOODHEAD*, S. R. CHAPMAN** AND J. M. NEWTON**†

*The Pharmacy Departments, *University of Nottingham, Nottingham, NG7 2RD, U.K., and **Chelsea College, University of London, Manresa Road, London SW3 6LX, U.K.*

The use of controlled sinusoidal vibration as a means of consolidating packings of lactose within small containers has been examined. Vertical vibration was found significantly more effective and reproducible than horizontal vibration in terms of the degree of consolidation achieved. An optimum frequency range was identified within which the densification was greatest, and this range was largely independent of particle size for particle size fractions of mean volume diameters ranging from 15.6 to 155 μm. The consolidation increased with increasing vibration acceleration up to a level beyond which no further decrease in porosity resulted. Typical effective vibration conditions were characterized by amplitudes of an order of magnitude similar to the particle sizes studied. For particle size fractions of mean diameters 17·8, 37·5 and 80·8 μm, there is evidence that an optimum particle size range exists, within which energy requirements for consolidation are at a minimum.

The packing density of bulk particulate solids can be increased by the application of sinusoidal vibration. Kinetic energy is imparted to the particles, which are then able to rearrange and find positions of closer packing. As a result, the porosity or void fraction of the bed is reduced, as it is when compression is applied. In contrast to the use of compression, however, vibration can reduce interparticle friction (Fisher & Coleman 1974) and shear strength of the powder bed (Roberts & Scott 1978).

The factors affecting the consolidation of powders by vibration may be seen to fall into two main categories, i.e. the properties of the powder (size, size distribution, shape, surface characteristics) and the vibration conditions (frequency, acceleration, amplitude and time).

The influence of particle size distribution has been widely discussed. Ayer & Soppet (1965, 1966) studied the vibratory compaction of spherical and angular particles, and related packing efficiency to the diameters of component particles. They found that packing could be optimized by combining selected size fractions; as the ratio of sizes increased, so did packing efficiency, up to a maximum value. Evans & Millman (1964) were also able to optimize packing in this way, but they suggested that the optimum size distribution would vary from one material to another. McGeary (1967), working with spheres, proposed that there should be at least a seven-fold difference between successive sizes to produce efficient packing. The effect of particle size itself is less clear, though Singhal & Dranchuk (1973), studying the vibratory compaction of several single component materials, mainly glass beads, found that large particles pack to a lower porosity than smaller particles.

There is disagreement over the influence of particle shape on vibratory consolidation. Shergold (1953) and Gray (1968) both suggested that angular or irregular particles can pack more densely than spherical ones when subjected to vibration. Ayer & Soppet (1965, 1966) and Evans & Millman (1964), on the other hand, found that packing efficiency increased with sphericity.

Interparticulate friction was discussed by Bell (1958) who stated that the greater the frictional interaction between particles, the greater the kinetic energy input required for consolidation.

Vibration parameters are inter-related and cannot therefore be considered entirely independently. At a given frequency, increasing the power input causes the acceleration (peak or RMS) and the amplitude of the vibration to increase. Conversely, increasing the frequency at a fixed power input results in a reduction in amplitude. The acceleration produced at a fixed power input is largely independent of frequency, but at the fundamental or resonant frequencies of a system under test, acceleration and amplitude are amplified. Hence several workers have advocated the use of resonant frequencies to

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† Correspondence.

optimize packing (Bell 1958; Evans & Millman 1964; Hauth 1967; Singhal & Dranchuk 1973). Such frequencies must normally be determined by trial and error. Shatalova et al (1967) put forward a rough guide for selection of frequency, based on particle size, while Evans & Millman regarded the range 20–5000 Hz as being useful for particulate materials. Hauth (1967), in assessing the packing of nuclear fuels, found that rapid cycling over a frequency range of several kHz, encompassing several resonant frequencies, was the most effective method.

The power input for vibratory consolidation should be within a critical range: if it is too low, the existing structure will be undisturbed, while excessive power may cause fluidization of the powder resulting in high porosity (Singhal & Dranchuk 1973). This is most likely at low frequencies, where the amplitude is relatively large. Stewart (1962) suggested that the amplitude of the applied vibration should be of the same order of magnitude as the size of particles being packed. The required power input, as would be expected, increases with the mass of the system.

The duration of vibration is a variable which has received little attention. Most workers quote times of several minutes to achieve the desired packing density, though Bell (1958) observed that in small samples, most of the consolidation occurs in the first 5 s. Larger and/or cohesive samples may require longer vibration times.

Gray (1968) pointed out that much of the work in this field was of an empirical nature, and could not be applied to systems other than those already studied. Spasskii (1977), however, has put forward a rationale for predicting suitable vibration conditions based on the known or measureable properties of a particulate system. His theory relates the required vibrational energy input to the energy characteristics of the particle contacts.

The purpose of this work was to study the effect of vibration conditions on the consolidation of a model particulate system having a controlled particle size.

MATERIALS AND METHODS

Materials

Crystalline lactose was chosen as a model powder system, and was classified into eight particle size fractions forming part of a $\sqrt{2}$ geometric progression, using methods described by Jolliffe (1980). The mean particle volume diameter of each size fraction was measured using a Coulter Counter (model TA, Coulter Electronics Ltd, Herts). Particle shape measurements were carried out with a computerized flying spot microscope (Eccles et al 1976). Angles of repose were known (Woodhead 1980), enabling the bulk flow properties of each size fraction to be assessed in terms of the classification of Carr (1970). The angle of internal friction of each fraction was determined by Jolliffe (1980) and found to be 36·5 ° for all the particle size fractions studied. Table 1 lists the measured characteristics.

Methods

All packings were prepared in identical cylindrical aluminium containers of internal diameter 19 mm and depth 44 mm.

Vibration was provided by one of two systems. The first consisted of a 5VA power oscillator and a model 790A vibrator (Goodmans Industries Ltd, Wembley, Middlesex). The second comprised a TA120 oscillator/amplifier, a VP3 electromagnetic vibrator and a portable vibration analyser (PVA) all supplied by Derritron Electronics Ltd, Hastings, Sussex. Provisions were made for attaching the cylindrical containers firmly to either of the vibrators, both of which could be operated in a horizontal or vertical position. A piezo-electric accelerometer (model A/01/T, D. Birchall Ltd, Mildenhall, Suffolk) was used in conjunction with the Derritron system to enable the vibration conditions to be monitored.

For the initial studies with the Goodmans system, the container was mounted centrally on the vibrator housing. For the Derritron System, Fig. 1, where an

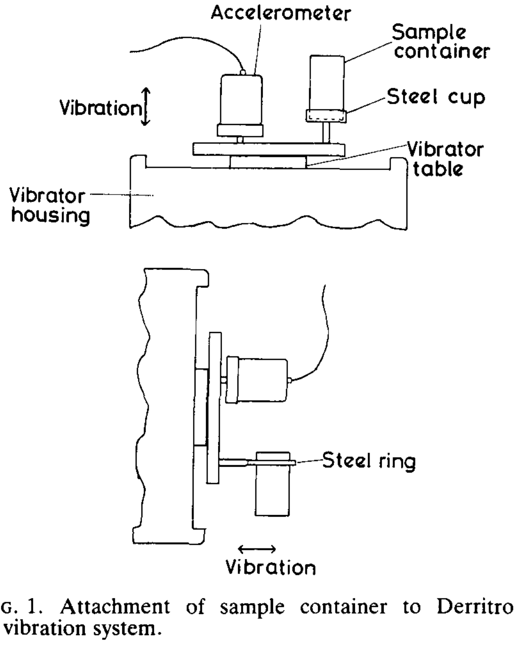

FIG. 1. Attachment of sample container to Derritron vibration system.

accelerometer was added to the system, the container and accelerometer were positioned to ensure balance of the two devices.

The general method of preparing the samples consisted of pouring powder into an aluminium cylinder until overfilled, then removing the excess with a spatula blade. Initial porosity was calculated from the weight of the packing, the volume of the container, and the apparent particle density of lactose previously determined to be 1·55 g cm^{-3} by an air comparison pycnometer (Beckman). The cylinder was attached, upright, to the vibrator, and a well-fitting cylindrical nylon rod 4 cm long was placed on the top of the packing to maintain a level surface during vibration. The consolidation produced by the application of vibration was determined by measuring the downward displacement of the rod with a travelling microscope, thereby allowing the calculation of the resulting porosity.

Identification of the useful frequency range

The Goodmans vibration system was employed to apply vertical and horizontal vibration to samples of lactose at a wide range of frequencies (0 to 10 000 Hz). From preliminary observations, the useful frequency range appeared to be between 30 and 600 Hz, irrespective of particle size, hence a number of frequencies in this band were investigated.

A sample of each particle size fraction was subjected to 30 s vibration at each frequency. The power output was not quantified, but was maintained at the same setting at each frequency (for all particle size fractions except the smallest, which required a higher power input in the horizontal mode to produce any measurable changes in densification.) Initial and final porosity values were recorded.

The measurement of amplitude at selected frequencies and accelerations

The Derritron vibration system was employed to apply vibration at four levels of acceleration, and at a number of selected frequencies, to packings of three particle size fractions, +18·7–26·5 μm, +37·5–53 μm and +75–105 μm. This was undertaken in both vertical and horizontal modes. The accelerometer and portable vibration analyser enabled the amplitude of vibration (0 to peak displacement) to be recorded under each set of conditions.

The influence of acceleration on porosity

In this experiment, the Derritron vibration system, incorporating the vibration analyser, was employed to apply vibration at increasing levels of acceleration to lactose packings, and to record the resulting changes in porosity.

Three size fractions, +18·7–26·5, +37·5–53 and +75–105 μm were chosen to provide a range of particle and bulk properties. For each of these, four frequencies in both vertical and horizontal modes were selected, which would produce the most consolidation, based on the results obtained in the previous experiment. At each frequency, a packing was vibrated for 10 min at an acceleration of 2 *g* (0 to peak). This was followed by 10 min at 3, 4, 5 *g* etc. until an increase in acceleration produced no further consolidation. The porosity achieved at each acceleration was recorded.

Reproducibility of vibratory consolidation

For each of the three lactose size fractions above, optimum horizontal and vertical vibration frequencies were selected. At each frequency, a vibration of 6 *g* acceleration was applied for 10 min to each of ten powder samples. Initial and final porosities were recorded, and a mean and coefficient of variation of the final porosity were calculated.

The preparation of samples in this experiment differed from the method described above, in that an aluminium collar was attached to the cylindrical container, effectively extending its depth from 44 mm to 72 mm. The packing was prepared and vibrated in this extended container, with the nylon rod in position as before. After vibration, the collar was removed and the remaining packing was levelled off using a spatula blade. The porosity could then be calculated from the weight of powder remaining since in all cases the consolidated packing completely filled the cylinder. By modifying the method in this way, any compression of the upper layers of the packing caused by the weight of the nylon rod would have little or no effect on the final packing, since the upper part of each packing was removed after vibration.

RESULTS AND DISCUSSION

The effect of applied vibration on the packing of particles is influenced by the ability of the particles to move relative to each other. The data presented in Table 1 indicate that there is no significant difference between the particle shape. The angle of internal friction of each of the particle size fractions was constant. Any difference in packing behaviour, therefore, will be governed by particle size and vibration conditions.

The effect of vibration frequency on consolidation is illustrated by Fig. 2 (a)–(d). For all eight particle

Table 1. Characterization of eight particle size fractions of crystalline lactose.

Designation of size fraction	Nominal particle size range (μm)	Mean particle volume diameter (μm)	Standard deviation (μm)	Mean particle* shape P^2/A	Angle of repose (°)	Flow properties
A	−18·7	15·6	11·1	—	60·6	Very cohesive
B	+18·7–26·5	17·8	9·4	17·4	51·6	Cohesive
C	+26·5–37·5	27·7	10·4	15·8	44·1	Fair to passable flow
D	+37·5–53	37·5	14·3	16·6	37·1	Free flowing
E	+53–75	41·3	14·8	16·0	34·6	Free flowing
F	+75–105	80·8	22·0	17·5	32·7	Free flowing
G	+105–150	99·5	34·7	19·5	32·6	Free flowing
H	+150	155·2	59·6	17·4	30·6	Free flowing

* P = perimeter. A = projected surface area.

size fractions, vertical vibration was more effective in reducing the porosity of the packings than horizontal vibration, over the frequency range studied. In all cases, there was a minimum in the curve, indicating an optimum frequency range. These minima varied in breadth, being relatively narrow in the case of the cohesive particle size fractions, and becoming broader for more free-flowing materials. The optimum frequency range was similar throughout the particle size range, suggesting that it may be more dependent on the overall size and shape of the sample/container system.

The minimum porosity achieved was approximately the same for each size fraction except the two most cohesive; these would require greater power input to reduce the porosity further. Some particle size fractions exhibited a second, sharper minimum at about 500 Hz in the horizontal mode. This may correspond to a resonant frequency for the system.

The relationship between acceleration and amplitude of vibration of the container at selected frequencies was independent of particle size and vibration mode. Therefore, each figure representing amplitude in Table 2 is the mean of six recorded values of displacement.

A progressive decrease in the porosity of packings occurred as the acceleration of vibration was increased up to a level beyond which no further

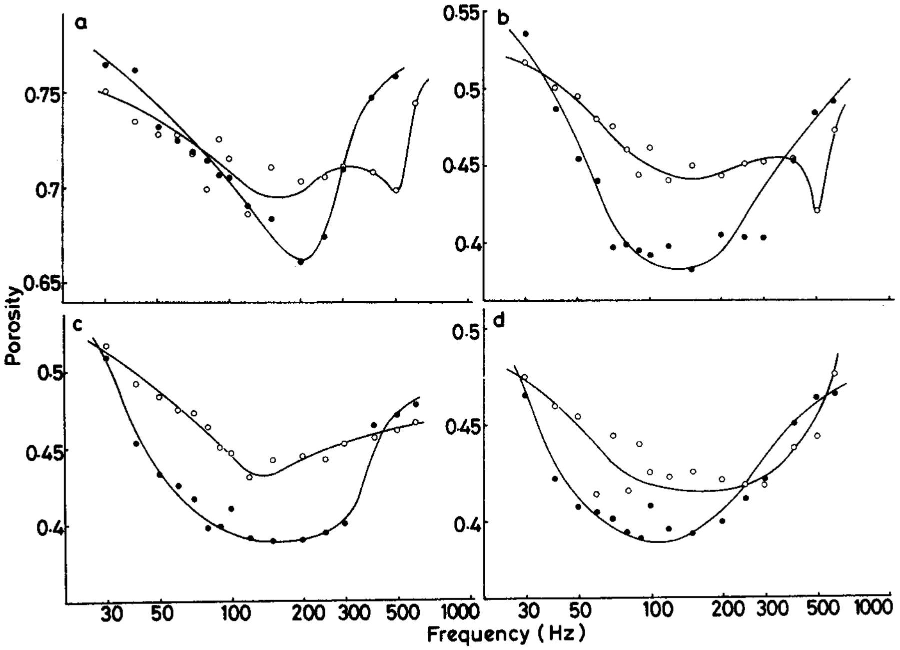

FIG. 2. The effect of vibration frequency on the porosity of a number of particle size fractions of lactose. ● Vertical vibration. ○ Horizontal vibration; (a) −18·7 μm; (b) +26·5−37·5 μm; (c) +53·0−75·0 μm; (d) +150·0−210 μm.

Table 2. Displacement (0-peak) in μm, recorded at various frequencies and accelerations.

Frequency, Hz	Acceleration (0-peak) in *g* 1	2	4	6
30	263	540	1083	1592
50	99	196	395	582
70	49	99	195	292
100	24	49	96	143
150	11·4	22	44	66
200	6·3	12·6	25	38
300	2·9	5·7	11·0	16·9
400	1·7	3·1	6·2	9·3
500	1·2	2·1	4·1	6·1
600	0·9	1·5	2·8	4·2

Table 3. Minimum porosities attained with lactose fractions B, D and F under various vibration conditions.

	Frequency (Hz)	Minimum porosity attained	Acceleration needed (*g*)
Fraction B, horizontal vibration	120	0·459	10
	150	0·466	11
	200	0·509	9
	250	0·468	11
Fraction B, vertical vibration	100	0·391	8
	150	0·381	7
	200	0·391	11
	250	0·397	10
Fraction D, horizontal vibration	100	0·392	9
	120	0·408	9
	150	0·410	9
	200	0·420	8
Fraction D, vertical vibration	70	0·390	5
	100	0·383	5
	150	0·385	6
	200	0·384	5
Fraction F, horizontal vibration	100	0·397	8
	120	0·404	10
	150	0·439	7
	200	0·415	12
Fraction F, vertical vibration	100	0·404	5
	150	0·395	7
	200	0·392	9
	250	0·392	8

densification occurred. Indeed, in several instances, the porosity began to rise slightly, due to excessive agitation of the packing which can disrupt the established arrangement of particles. Fig. 3 includes an example of this behaviour for particles vibrated at 200 Hz. Table 3 lists the minimum porosities attained, together with the acceleration required in each case. With all size fractions, vertical vibration generally produced the greatest reduction in porosity, and at lower accelerations than those required in the horizontal mode. By referring to Tables 2 and 3, it can be seen that, in general, typical optimum conditions, of 100–200 Hz vibration at an acceleration of 6 *g*, are characterized by amplitudes of the same order of magnitude as the particle size range of the material under test. This supports the work of Stewart (1962), who suggested that the vibration amplitude should be similar to the particle size. On this basis, larger particle sizes would require greater amplitudes and therefore greater acceleration at a given frequency to produce minimum porosity. This appears logical since the particles of greater mass will require a higher energy input to overcome gravitational forces and disrupt the existing packing.

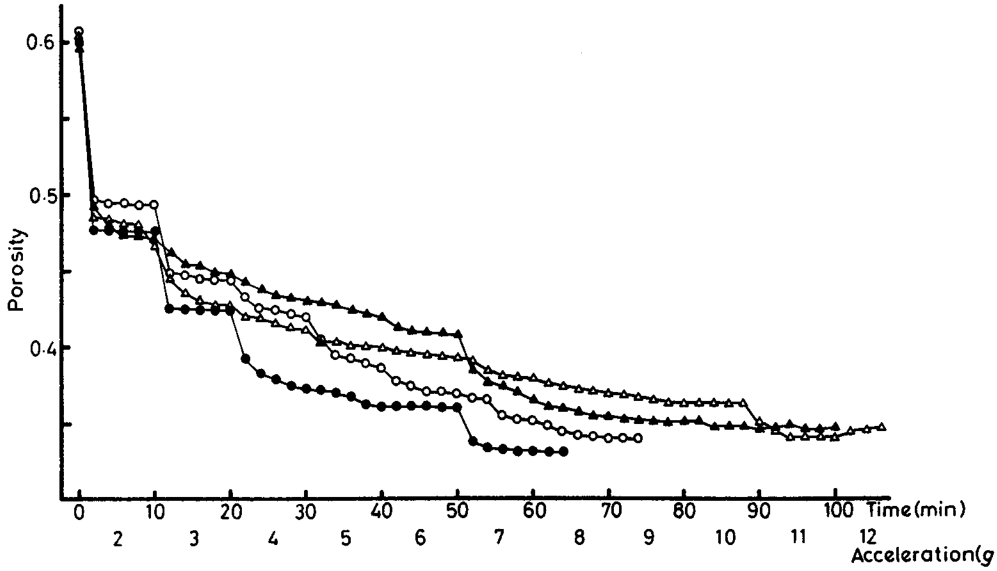

FIG. 3. The effect of increasing the acceleration of vertical vibration on the porosity of lactose, particle size +18·7−26·5 μm. Vibration frequency: ○ 100 Hz; ● 150 Hz; △ 200 Hz; ▲ 250 Hz.

Conversely, for smaller particles interparticulate forces will also tend to oppose the relative motion of adjacent particles. Thus, there may be an optimum particle size range for a given material, within which the material is most susceptible to densification by vibration. The acceleration values in Table 3 indicate that of the three size fractions of lactose studied, the intermediate size, +37·5–53 μm, requires least energy input to achieve minimum porosity. This size fraction, being the smallest of those described as free-flowing, appears to be in the optimum range for consolidation by vibration.

The reproducibility of vibratory consolidation at selected frequencies and accelerations is shown in Table 4. Coefficients of variation of final porosity were consistently lower for vertical vibration than for horizontal vibration, and as observed in previous experiments, vertical vibration produced packings of lower porosity.

Table 4. The porosity and its variability of lactose samples subjected to vibratory consolidation at an acceleration of 6 *g* for 10 min.

Lactose size fraction	Vibration mode	Vibration frequency (Hz)	Mean porosity	Coefficient of variation (%)
B	Horizontal	120	0·500	2·16
B	Vertical	150	0·478	1·33
D	Horizontal	100	0·441	1·23
D	Vertical	100	0·404	0·67
F	Horizontal	120	0·477	1·65
F	Vertical	150	0·408	1·06

Thus it has been established that in the preparation of powder beds with minimum, uniform porosity, vertical vibration at optimum conditions of frequency and acceleration is required. This has practical implications in the selection of minimum container sizes for powders and the preparation of powder beds suitable for the removal of uniform samples, as for example the use of dosator nozzles to fill hard gelatin capsules.

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