Dynamic Measurement for the Stiffness of Loosely Packed Powder Beds

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An experimental methodology to measure the stiffness of loosely packed powder beds was developed, which had not been possible using previous methods. Experiments were performed on a range of sample powders including polyethylene, rubber, glass, sand, and clay powders. Powders were placed in a vertical, open, cylindrical vessel, and subject to sweep vibration at low accelerations. Base force and acceleration were measured using an impedance head and accelerometer. Apparent mass, defined as a ratio of base force to base acceleration, measured showed a significant peak frequency. The longitudinal elastic modulus of the bed was calculated from the data. For shallow beds in which the wall friction is negligible, the peak frequency is independent of the cross-sectional area of the bed and sweep rate, but dependent on the bed height and acceleration. Data generated by the top-cap method agreed reasonably for cases in which the packing state was not sensitive to external force. A substantial change in elasticity was detected with changes of packing states. Furthermore, the elasticity of packed beds conforms to Kendall's fourth-order relationship with solid volume fraction over a range of packing conditions.

Introduction

In most powder processing, powders exhibit behavior that is neither completely solidlike nor completely liquidlike, but intermediate between the two. One of the factors causing such a behavior may be the ability to sustain different degrees of packing (Fayed and Otten, 1997). The dynamic response and properties of powder beds are also critically dependent upon the packing state (Das, 1993; Shinohara et al., 2000). Various tests including uni- or triaxial compression tests and others have been used in order to measure the stiffness properties of powder beds (such as Alsop et al., 1995, 1997; Bassam et al., 1991; Bika et al., 2001; Das, 1993; Matchett and Alsop, 1995; Matchett et al., 1998, 1999, 2000). In such tests, the sample powders have to be consolidated prior to the measurement, resulting in high packing fraction of the bed (Alsop et al., 1995, 1997). However, powder processes are often concerned with powder beds packed relatively loosely rather than with such dense beds.

Okudaira et al. (Okudaira et al., 1993; Okudaira, 1997) have studied extensively the dynamic properties of loosely packed powder beds using the sound of white noise, indicating that it is possible to determine the longitudinal elastic modulus of the bed from the sound absorption peak frequency for powder beds comprising relatively small-size particles. This method can apply to beds packed naturally by only gravity,

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but not for systems comprising relatively large-size particles, due to the sound absorption mechanism. For this reason, they (Okudaira et al., 1994) proposed the top-cap method (TCM) in terms of acceleration transmissibility through shallow beds using the top-cap mass that surmounted the bed surface; in principle, TCM can be applicable for all powders. Unfortunately, TCM requires the preconsolidation of a sample prior to the measurement, because the powders have to support the top-cap mass, resulting in relatively denser beds compared to a natural packed bed, especially for powders in which the packing characteristics are sensitive to external force, that is, the Hausner ratio is high.

Kendal et al. (1987a,b) reported theoretically and experimentally the effect of the packing fraction upon the elasticity of particle assemblies. Their theory appeared to successfully describe the experimental values of elastic modulus for particle assemblies, and in particular the strong influence of particle packing, which was theoretically predicted to be the fourth power of the solid volume packing fraction. They adopted the use of a three-point bend method to measure the elastic modulus, reporting that only a relatively narrow range of packing fractions could be tested, because the powder became excessively fragile at low packing fractions. Considering the structural principle of the three-point bend, it would be thought that the natural packed state might be much lower than the range investigated. As far as we are aware, no relevant system exists for measuring such a loosely packed bed.

This article presents an experimental methodology to measure the stiffness of loosely packed powder beds using a sweep vibration at low accelerations in terms of vibrating apparent mass. Applying such low accelerations enables the stiffness of the loosely packed bed to be measured. The apparent mass, defined as a ratio of base force to base acceleration, is measured as a function of frequency. The effects of experimental variables upon the apparent mass data are systematically examined. It is possible to derive the longitudinal elastic modulus of the bed from the apparent mass data, and the data are discussed and compared with data generated by TCM.

Experimental Studies

Experimental apparatus and procedure

The experimental system developed herein is structurally similar to the TCM developed by Okudaira et al. (Okudaira et al., 1994; Okudaira, 1997); however, the principle is completely different. This system is able to perform without a top-cap mass, which enables the properties of loosely packed beds to be measured. Figure 1 illustrates the experimental system. A Perspex cylindrical test cell was mounted on an impedance head, consisting of a force transducer, driven by an electromechanical vibrator, with a vertical line of action. To investigate the effect of the vessel shape, the test cell size was set between 0.01 and 0.08 m for the vessel height and 0.0345 and 0.149 m for the vessel inner diameter. The vibration exciter was placed on an isolator platform to minimize other sources of noise from the general surroundings. Preliminary tests demonstrated that the background noise was found to be negligible within this study. The vibrator was a Ling system with a programmable feedback controller. Feedback control was achieved via the accelerometer on the base plate, which provided input signals for the controller to modify and provide output feed to the power amplifier that drove the vibrator, to give the required acceleration at a chosen frequency range. The accelerometer and force transducer were of the piezoelectric type and made by Kistler. They were connected via a coupler to a CED, A/D interface, which recorded data at a rate of 12.5 kHz per channel onto the hard disk of a PC. The sampling frequency was larger than the minimum

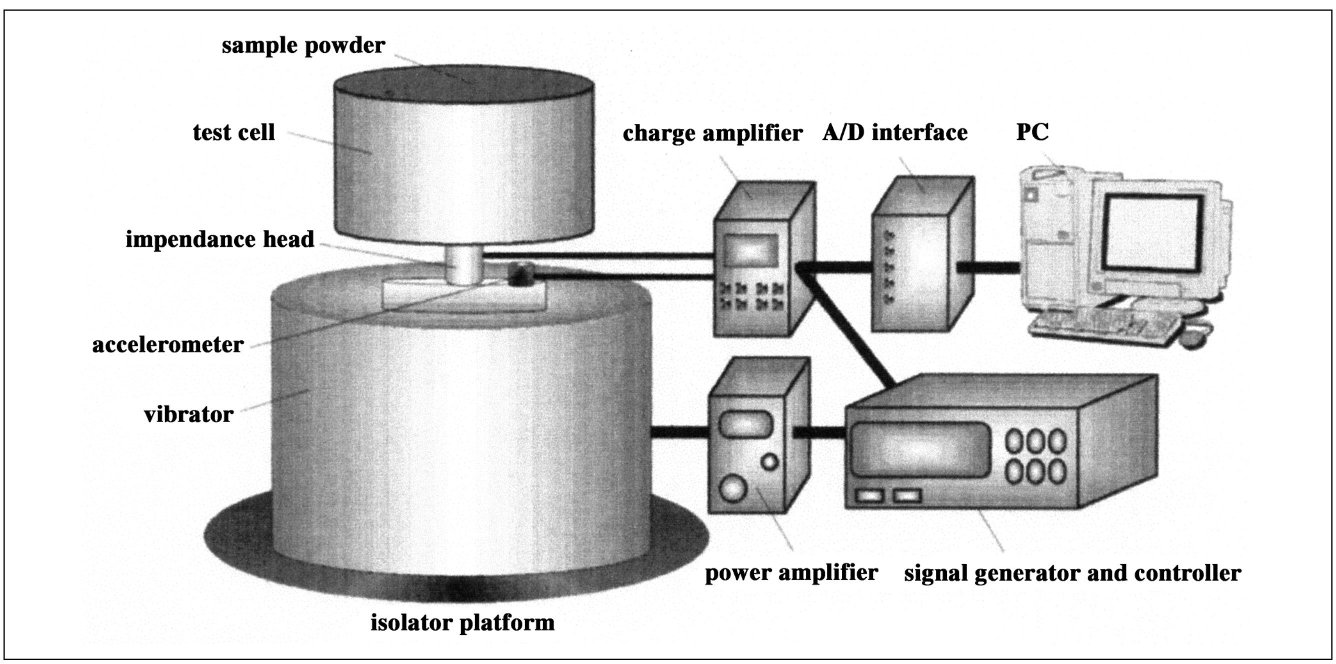


Figure 1. Experimental system developed.

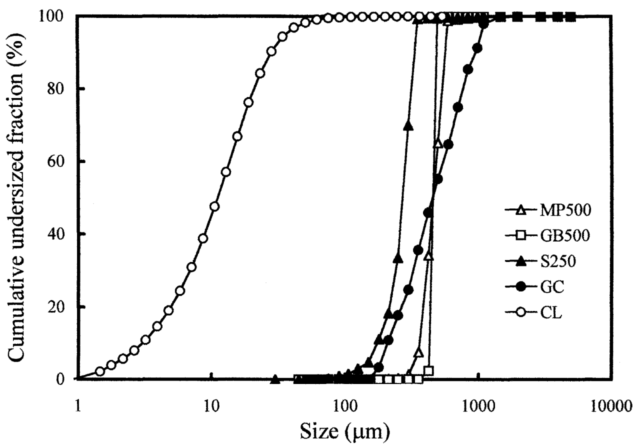


Figure 2. Size distributions of sample powders used.

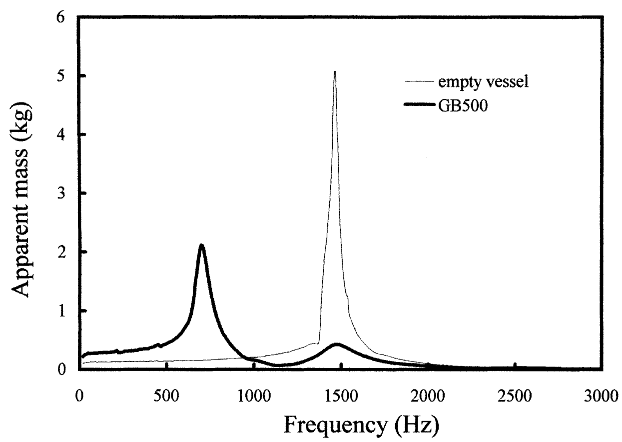


Figure 3. Typical data of apparent mass measured as a function of frequency.

sampling frequency, that is, Nyquist frequency, which was 8 kHz within the range of this study, based upon Shannon's sampling theorem (Norton, 1989). The piezoelectric devices are characterized by very rapid response times with high accuracy and reproducibility, providing very reliable data acquisition.

The sample powders were placed carefully into the test cell by gravity using a spatula and subsequently leveling the surface of the bed with a straight edge. Then the sweep vibration that ranged from 10 Hz to 4,000 Hz was applied to the test cell through the base at the chosen sweep rate in the 0.833–15-Hz/s range with a constant peak acceleration that ranged from 0.196 m/s^2 to 7.35 m/s^2. Within this frequency range, the vibrator, force transducer, and accelerometer were precise and independent of their structural resonance. During the sweep, data from two measurement devices were monitored as a time series on the hard disk of the computer. The collected data points for each channel were ranged from 3.3×10^6 to 3.0×10^8 depending upon the sweep rate. Subsequently, these time-series data were analyzed by fast Fourier transform (FFT) with a sampling number of 4,096 points to give the frequency data. An apparent mass, defined as a ratio of base force to base acceleration, was calculated by the computer. To ensure the reproducibility of tests, each test was repeated three times. The deviation upon the bulk density for each test was within plus/minus 10% over a range of this study, which is generally referred to in the literature as a loosed bulk density (Fayed et al., 1997).

Sample powders

Experiments were performed on low-density polyethylene powder, glass spheres, building sands, rubber powder, and clay with the size distributions shown in Figure 2. The size data of clay was measured by a laser diffraction analyzer (MSIZER, Malvern Instruments Ltd.) and the other data were obtained by the sieve method. The physical properties of the sample powders are given in Table 1 (Lee et al., 1983; Waterman and Ashby, 1997). The polyethylene powder, glass sphere, and sands were sieved to provide materials with narrow size ranges. The loose bulk density was measured by pouring a known weight of material through a standard funnel into a measuring cylinder. This was then compacted by lifting the cylinder and allowing it to drop from a fixed height until no observable change in bulk density occurred, in order to measure the tapped bulk density.

Results and Discussion

Apparent mass as a function of frequency

Figure 3 shows typical data of apparent mass measured as a function of frequency. Data of GB500 was plotted in the figure along with the empty-vessel data. The cylindrical vessel had an inner diameter of 0.08 m and was 0.02 m high. The acceleration level and sweep rate were 0.196 m/s^2 and 5 Hz/s, respectively. Prior to discussing data for powders, it would be useful to note the dynamic response of the empty vessel. Data for the empty vessel showed a sharp peak around 1500 Hz, corresponding to the resonance of the vessel. Also, the vessel resonance varied with its dimension. Following from the definition of apparent mass, the mode shape is considered to be the first mode in which the node is positioned at the center of vessel base, which is fixed by the impedance head.

Table 1. Physical Properties of Sample Powders Used

Materials (key)	Mean Size (μm)	True Density (kg/m^3)	Loosed Bulk Density (kg/m^3)	Tapped Bulk Density (kg/m^3)	Hausner Ratio
Polyethylene powder (MP500)	459	918	339	406	1.198
Glass sphere (GB500)	462	2,500	1,549	1,623	1.048
Sand (S250)	271	2,600	1,384	1,593	1.151
Grey crumb powder (GC)	382	1,000	191	297	1.555
China clay-kaolin (CL)	10.5	2,640	367	670	1.826

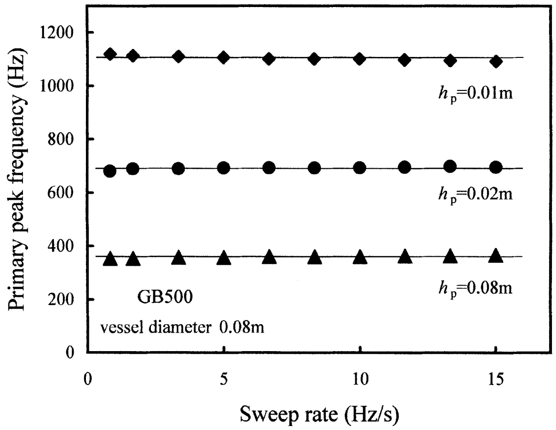


Figure 4. Effect of sweep rate upon the peak frequency.

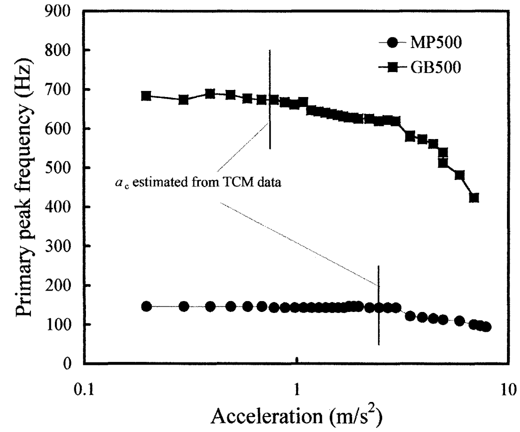


Figure 5. Effect of acceleration upon the peak frequency.

From Figure 3 it can be seen that the presence of the powder exhibited a significant peak, which appeared at a much lower frequency range than the vessel resonant frequency. This was observed over a range of vessel dimensions and sample powders. In such a low-frequency range, it is obvious that the vessel behaves almost as a rigid body. Consequently, the peak exhibited by the powder is simply due to the bed resonance. In addition, the harmonic resonance of the loosely packed bed is consistent with implications reported by Okudaira et al. (1993) using the sound-absorption technique. They demonstrated that the bed behaved as a single harmonic resonator at low acceleration. Furthermore, using a very small accelerometer they showed that the amplitude profile within the bed for the longitudinal direction obeyed to theoretical prediction based upon a single harmonic vibration mode. There are several factors that may affect the peak frequency, for example, the effects of vibration variables and the vessel shape. Let us examine these effects in the following sections.

Effects of vibration variables

Since we adopt the use of sweep vibration, the effects of vibration variables upon the peak frequency are examined by changing the sweep rate and acceleration level. Figure 4 shows the effect of sweep rate upon the peak frequency. GB500 was used as a sample powder, and the acceleration level was 0.196 m/s^2. The peak frequency was found to be insensitive to the sweep rate for the low acceleration level, indicating the linearity of this system. This allows stochastically the use of white noise and/or impact (Okudaira et al., 1994). In the following experiments, we used a sweep rate of 5 Hz/s.

Figure 5 shows the effect of the acceleration level upon the peak frequency. GB500 and MP500 were used in the experiments. Within the range of accelerations used, the macroscopic particle packing did not show any changes during measurement. The peak frequency is almost constant unless the acceleration exceeds a certain critical level, a_c, that was dependent upon the sample materials. Above a_c, a decreased peak frequency was found. This trend is quite similar to results of Okudaira et al. (1994) using the TCM, but they gave only a qualitative explanation regarding the decreased stiffness in terms of the changes in particle contacts due to the increased acceleration. Here, we attempt to give a quantitative interpretation for the decreased stiffness. Presumably, a_c can be associated with the bed resonance, because the amplification due to the resonance can cause the instantaneous breaking of particle contacts even below 9.8 m/s^2 of the base acceleration. Therefore, a_c is thought of as the critical base acceleration at which the bed surface acceleration exceeds gravity acceleration, due to the elastic amplification effects. Obviously, the maximum amplification occurs at the resonant frequency f_r. Then, a_c is given as

$$a_c = \left[\text{gravity acceleration}/T(f_r)\right] \tag{1}$$

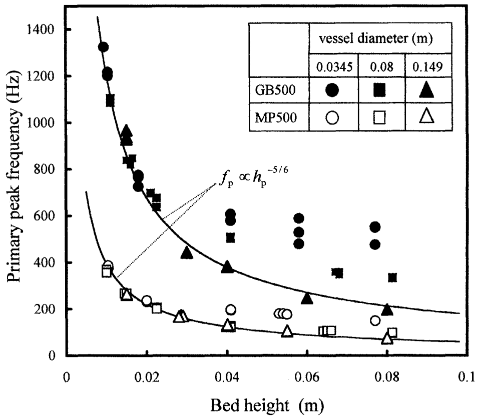
where $T(f_r)$ is the maximum amplification value at the resonant frequency. Here, $T(f_r)$ is given in terms of the effective mass, m, stiffness, k, and damping, c, which are measurable using the TCM, as follows (Yanagida et al., 2001a,b; 2002a,b)

$$T(f_r) = \left\{ \frac{\left[k^2 - (2\pi f_r)^2 mk + (2\pi f_r)^2 c^2\right]^2 + \left[(2\pi f_r)^3 cm\right]^2}{\left\{\left[k - (2\pi f_r)^2 m\right]^2 + (2\pi f_r)^2 c^2\right\}^2} \right\}^{1/2} \tag{2}$$

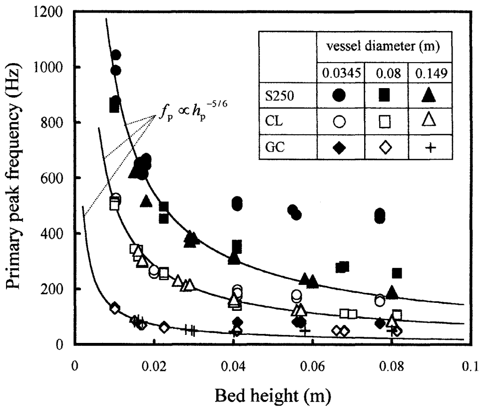
Alternatively, $T(f_r)$ can be directly measured from the experimental data of TCM. The calculated a_c is given in Figure 5, in a reasonable quantitative agreement with the experimental trend. In agreement with the purpose of this study, the following experiments were performed with an acceleration of 0.196 m/s^2, well below the critical acceleration level.

Effect of vessel shape

Wall friction is an inherent problem in powder characterizations (Fayed et al., 1997). In order to develop a relevant powder testing system, the wall effects have to be justified. Ideally, on the system developed herein, the wall effect should



(a)



(b)

Figure 6. (a) Effect of vessel dimension upon the peak frequency; (b) effect of vessel dimension upon the peak frequency.

be negligible, because this system aims to extract solely the bed stiffness from the measured apparent mass data. Theoretically, if the wall friction were negligible, the peak frequency should be insensitive to the cross-sectional area and dependent only upon the height.

Figure 6 shows the measured peak frequency as a function of bed height for three types of inner diameters ranging from 0.0345 m to 0.149 m. The peak frequency experienced a clear difference between samples, reflecting the different material properties. In all configurations, the peak frequency decreases with an increase in the bed height. For deep beds, there is a significant difference between the different diameters, indicating wall effects. On the other hand, for shallow beds, the peak frequency appears to be independent of the diameter and dependent only upon the bed height. Theoretically, the peak frequency is proportional to the inverse of the height for an ideal elastic system and to the power of $-5/6$ of the height for Hertzian contact. The data conformed to the Hertzian prediction, indicating nonlinear contact stiffness. The insensitivity of the diameter and the dependence of the height upon the peak frequency allow us to use the shallow-bed system without wall effects showing any significant involvement.

Table 2. Y and v_p Estimated from the Peak Frequency

	Y (Pa)	v_p (m/s)	ϕ
MP500	1.46×10^5	20.6	0.377
GB500	5.76×10^6	61.2	0.616
S250	3.66×10^6	51.4	0.534
GC	1.18×10^4	7.83	0.192
CL	2.51×10^5	26.4	0.137

Determination of longitudinal elastic modulus and comparison with TCM data

The preceding results showed that (1) the correspondence between the peak frequency and the bed harmonic resonance, and (2) the effects of experimental variables upon the peak frequency, identifying appropriate experimental configurations. Based upon these findings, we attempt to determine the longitudinal elastic modulus of the bed, Y, from the measured peak frequency as follows. Here, we consider the bed dynamics in the longitudinal direction. Since the mode shape is considered to be the one-end-fixed longitudinal mode, the velocity of the longitudinal stress wave propagation through the bed, v_p, is given in terms of the peak frequency, f_p, and the bed height, h_p, as follows (Das, 1993; Okudaira et al., 1993)

$$v_p = 4h_p f_p \tag{3}$$

If v_p is expressed as $\sqrt{Y/\rho_b}$ (ρ_b is the bulk density of the bed), then Y is given by

$$Y = \rho_b(4h_p f_p)^2 \tag{4}$$

Consequently, Eqs. 3 and 4 enable Y and v_p to be quantified from the measured f_p along with h_p and ρ_b, which are also experimentally measurable. However, it should be noted that the calculated Y is a little larger than the actual elasticity of the materials, because the present system does not allow the lateral expansion of the samples due to the presence of the vessel wall. Generally, Y is referred as a constrained modulus (Das, 1993). Also, the formula assumes no damping in the system, and this would be a reasonable approximation because the damping ratio was found to be very small (Das, 1993; Okudaira et al., 1994, 1997; Yanagida et al., 2001a,b).

Table 2 shows the calculated data of Y and v_p with the packing fraction. These experiments were performed with the vessels of low aspect ratios, below 0.5. For comparison, Table 3 shows data generated by TCM with a top-cap mass of 0.096 kg. For GB500, whose packing state was relatively insensitive to external force, a reasonable agreement between the pre-

Table 3. TCM Data

	Y (Pa)	ϕ
MP500	4.46×10^5	0.442
GB500	7.74×10^6	0.626
S250	5.91×10^6	0.579
GC	1.11×10^5	0.280
CL	8.93×10^5	0.196

sent measurement and TCM data was seen. Although TCM data were slightly larger than the present measurement, this could be interpreted in terms of the nonlinear contact stiffness by the presence of top-cap mass (Okudaira et al., 1994). On the other hand, a substantial discrepancy was found for other samples with changes in the packing fractions. Especially, the data of GC were an order of magnitude smaller than TCM data. This could be mainly due to the effect of the packing conditions. As seen in Table 1, the packing states of these sample powders were quite sensitive to external force, resulting in relatively high packing fractions in TCM data. However, it should be noted that the resultant high packing fraction is still much lower than the range required for a conventional unconfined compression test (Mcglinchey et al., 1997; Richardson et al., 2000). The result indicates that the effect of the packing condition upon the bed elasticity is not negligible and even the preconsolidation required for TCM gives changes in the packing fraction. This will be considered in relation to Kendall's fourth power-scaling law, as discussed in the next subsection.

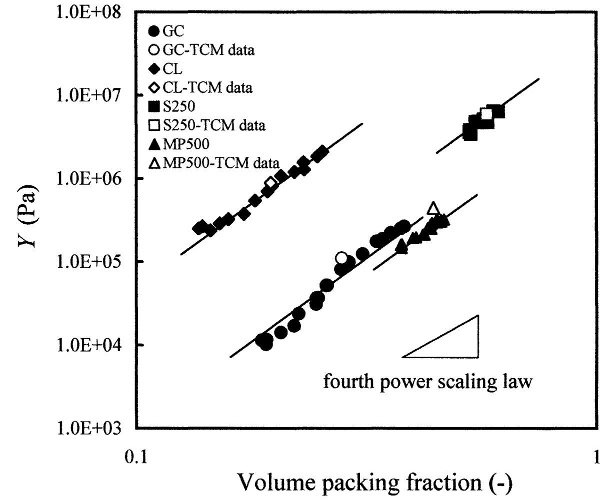


Figure 7. Longitudinal elastic modulus as a function of volume packing fraction.

Vessel inner diameter 0.149 m, bed height 0.04 m.

Effect of packing condition

The effect of the packing condition upon the bed elasticity is now examined. Kendal et al. (1987a,b) considered the ensemble elastic modulus of a particle assembly to be dependent on the contacts between individual particles, and proposed the following mathematical relationship between the effective elastic modulus, Y, the material elastic modulus, Y_t, the interfacial energy of the solid particles, Γ, the volume packing fraction of solids, ϕ, and the particle diameter, D

$$Y = 17.1\phi^4 \left[\frac{\Gamma Y_t^2}{D}\right]^{1/3} \quad (5)$$

This formula implies the very strong influence of particle packing through the fourth-order relationship. Furthermore, Kendall et al. demonstrated that the fourth power scaling law was applicable for titania, zirconia, silica, and alumina powders whose particle sizes were submicron (Kendal et al., 1987a,b). Since the size range used here is much larger than those, it would be interesting to investigate the applicability of their formula to such large particles.

In order to manipulate the packing condition, vibration and compaction by an iron disk of 1.59 kg were adopted. The applied vibration was 50 Hz, and the acceleration levels ranged from 9.8 m/s^2 to 29.4 m/s^2. After pouring a known weight of samples into the test cell, preconsolidation was performed until no observable change in bulk density occurred. This was repeated until the samples filled the test cell, using a straight edge to level the bed as necessary. The preconsolidation was carried out carefully to ensure a homogeneous mass distribution within the bed. Figure 7 shows Y as a function of the packing fraction. The vessel dimensions were 0.149 m inner diameter and a height of 0.04 m. The results of all samples gave a reasonable agreement to the fourth power-scaling law over a range of packing fraction. In addition, TCM data were consistent with the data of consolidated samples for an identical packing fraction. As reported by Kendall et al., the fourth power-scaling law can be understood in terms of two factors: the density of particle packing and the coordination number of each particle. However, the agreement raises an interesting question regarding the effect of size distribution, because the theory may become less reliable if the particles are not uniform in size (Kendal et al., 1987b). For instance, data of CL agreed well with the scaling law in spite of the relatively wide size distribution that ranged from 1 μm to 100 μm, as shown in Figure 2. This also applies to the data of GC. Surprisingly, the fourth power-scaling law was observed in weakly attractive colloidal suspension systems, as reported by Trappe et al. (Trappe and Weitz, 2000; Trappe et al., 2001). Although it could be inferred that there is an underlying theory that governs the relationship between the bed elasticity and the size distribution, further theoretical and experimental works need to be undertaken in this area.

Adapting Eq. 5, it is possible to estimate Γ from data of Figure 7. For example, the interfacial energies of S250 and GB500 were found to be 1.38×10^{-6}, 2.36×10^{-6} J/m^2, respectively. Although the materials were different, these values are at least four orders of magnitude lower than Kendall et al.'s value of pure silica—0.05 J/m^2 (Kendal et al., 1987b). Thornton (1993) pointed out that the coefficient of Eq. 5, 17.1, was overestimated and should be 10.26 for a simple cubic array and 7.62–8.00 for a body-centered cubic array, which is dependent upon Poisson's ratio of the particles. However, adapting these lower coefficients, the estimated value of interfacial energy was still found to be much lower, for example, 6.40×10^{-6} J/m^2, with a coefficient of 10.26 for S250. Although there are several possible reasons for this, such as the particle-surface contamination, the particle size and/or shape, and others, further investigations are required.

The results in Figure 7 highlight that,

(1) The discrepancy in Tables 2 and 3 can be interpreted as the effect of the packing condition;

(2) The developed experimental methodology can give the stiffness of loosely packed beds, which has not been possible using earlier methods.

As a typical example, Figure 8 illustrates the range and applicability of the developed system, TCM, and an unconfined compression test (Richardson et al., 2000) in the case of CL.

Conclusions

An experimental methodology to measure the stiffness of loosely packed powder beds was developed by applying a sweep vibration at low accelerations. Apparent mass data as a function of frequency showed a significant peak corresponding to the resonance of the bed. For shallow beds in which the wall effect is negligible, the peak frequency is independent of the cross-sectional area of beds and the sweep rate, but dependent on the bed height and acceleration level. The peak frequency gives the longitudinal elastic modulus of powder beds via the velocity of longitudinal stress-wave propagation. Comparison of these data with data generated by TCM showed a reasonable agreement for the cases in which the packing state was not sensitive to an external force, while a substantial change was observed with the changes in the packing states. A series of investigations of the effect of the packing fraction upon stiffness showed that the elasticity of packed beds conforms to the fourth power-scaling law over a range of packing conditions. These results demonstrate that the experimental methodology developed herein is very useful to measure the stiffness of loosely packed beds, which has not been possible using earlier methods. This method has been compared to the TCM with satisfactory results. Okudaira et al. (Okudaira et al., 1994; Okudaira, 1997) successfully compared the TCM to a sound absorption method. However, it remains for further investigations to compare this method to dense particle-bed methods, such as unconfined compression and three-point bend methods.

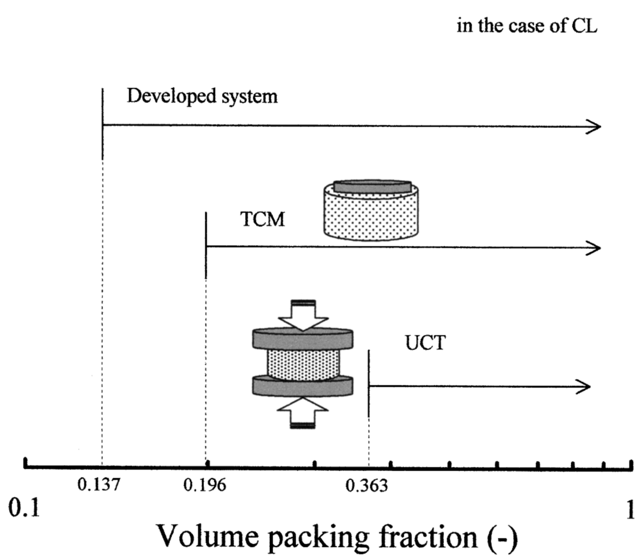


Figure 8. Range and applicability of the developed system, TCM and UCT (unconfined compression test).

Acknowledgments

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