The Dynamic Measurement of the Physical Properties of Powders Using Vibration – the Mixing Effect of a Small Amount of Fine Powder

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Abstract

A vibrational tester for measuring the dynamic physical properties of powders has been developed. The tester system consists of an electromagnetic vibrator with a controller for frequency and acceleration, a solid sphere sensor attached to a load transducer, and a microcomputer.

It has been shown that the characteristic curves, when measured as the relationship between the frequency and the vertically transmitted force through the powder bed, are sensitive to changes in the physical properties of the powders. The mixing effect of a small amount of fine powder on the change in the peaks of the characteristic frequency has been measured using two different mixers.

1. Introduction

Some physical properties of powders, such as flowability and friction coefficients, are not solely the result of the physical and chemical properties of the particles themselves, but are also due to the dynamic conditions created by external forces such as gravity, vibration or electromagnetism. Vibration has been applied in practical processes (e.g., conveying, packing and distributing powders) and has also been used to study the fundamental characteristics of powder beds.

In previous work 1), whose objective was to evaluate powder flowability as a function of applied vibrations, an apparatus for measuring the resistance acting on a solid sphere inserted into the powder bed and horizontally displaced while the bed is vibrated was developed and tested. The influence of the vibration frequency on the fluidization and packing characteristics of the powder bed was investigated, and, as an application, the optimum frequency to be used in vibration-aided mixers could be selected. The same paper also considered the possibility of using this measuring apparatus to detect slight changes in the flowability of the powder by measuring simultaneously the powder's resistance to the horizontal movement of the solid sphere, and the vertical force transmitted through the vibrating bed onto the sphere. For this purpose, another load cell (for measuring the vertically transmitted force) has been included in the system. The horizontal motion of the sphere has also been improved by using a sliding plate to effect displacement.

With this new measuring system, experiments have been conducted first to obtain the relationship between the frequency of the applied vibration and the vertically transmitted force and, second, to study the effect that adding small amounts of fine powders will have on the peaks at the characteristic frequencies. For the latter, two different mixers were used and the resulting characteristic curves were compared.

2. Experimental Equipment and Method

Figure 1 is a schematic diagram of the vibratory powder tester. Basically, the tester consists of a cylindrical vessel (13) mounted on an electromagnetic vibrator (14) and a solid sphere sensor (11) suspended into the vessel by a supporting rod (12) attached to the upper part of the equipment. As described in the pre-
As the vibration frequency is varied, the acceleration is maintained at the desired value by controlling the vibration amplitude. The mechanical force exerted by the powder on the sphere is converted into DC potential by means of two load transducers placed under the sliding plate. One load transducer measures the vertical compression force and the other measures the horizontal compression force as the sphere is displaced.

In a previous evaluation of the relationship between the force and the corresponding output voltage, the measuring system was found to be able to follow up the frequency sweeping rate. By means of a variable-speed motor (8), the sphere can be moved along a distance of 0.05 m, and its displacement is measured by a differential transformer (4).

Two pressure sensors are also included in the equipment, one attached to the sphere, the other to the bottom wall of the vessel containing the sample. The sphere can be initially placed into the vessel at a depth of approximately 0.1 m. The data supplied by all the sensors is collected by a multichannel data logger (22) (Brain IV, 12 bit, 16 ch) and processed by a microcomputer (23).

Some representative physical properties of the powders tested in this work are shown in Table 1. Before testing, the alumina powder was washed with acetone and dried.

The tested mixtures were prepared using two different mixers, namely, (I) a high-speed stirring mixer² (SFC-5, Kawata Ltd.) and (II) a rotary drum with simultaneous rocking motion³ (RM-10G, Aichi Electric Co.). With mixer (I), three different mixtures were prepared: i) a wax with a low melting point was added to alumina and stirred until the frictional heat thus generated raised the temperature of the mixture above the wax's melting point, resulting in the adhesion of fine wax particles onto the surface of the alumina particles⁴; ii) BN (boron nitride) was added to the mixture obtained in i); iii) BN was directly added to alumina. All these mixtures were prepared with an impeller agitation speed of 1500 rpm while the mixer was vibrated at 41.7 Hz.

With mixer (II), mixtures (of two components) were prepared by rotating the vessel at 63.3 rpm with a rocking speed of 10 min⁻¹ (rocking angle 20°) for one hour.

Besides the measuring conditions, other factors likely to influence the vibrating state of the bed include the characteristics of the solid sphere and the vessel itself. By fixing the measurement conditions and using a reference powder for comparison, the method for evaluating the relative variation of the physical properties of different powders was tested.

The measuring conditions used in these experiments are listed in Table 2. For measure-
Table 2 Measuring conditions of the powder tester

| Parameter                             | Value          |
|---------------------------------------|----------------|
| Diameter of cylinder                  | 0.08 [m]       |
| Height of cylinder                    | 0.19 [m]       |
| Height of powder bed                  | 0.08 [m]       |
| Diameter of sphere                    | 0.02 [m]       |
| Position of sphere center             | 0.035 [m] from the bottom center of the cross-section |
| Range of frequency                    | 20 ~ 440 [Hz]  |
| Sweeping rate                         | 1.6 [Hz/s]     |
| Magnitude of acceleration             | 1G             |

ments, a fixed quantity of the sample is introduced into the testing vessel, and the sensing sphere is inserted and placed at a prescribed position within the bed. Then, to fix the initial packing state for all samples, the particle bed is vibrated sinusoidally at 60 Hz and at an acceleration of 1G for 10 minutes. Once this preliminary conditioning of the bed is accomplished, the initial frequency is set, and the frequency range is swept at a fixed acceleration of 1G. At the same time, the vertically transmitted force $F_V$ at each frequency is measured continuously.

3. Results and Discussion

3.1 Relationship between the vertically transmitted force $F_V$ and the frequency $f$

Figure 2 shows the output curves of the vertically transmitted force as a function of the frequency and the sweeping rate of the frequencies obtained for alumina at frequencies between 20 ~ 450 Hz. It can be seen that the fine details of the output curves are lost as the sweeping rate increases because it takes a finite time for the powder bed to attain a steady state when it is vibrating at a given frequency. If the sweeping rate is high before the bed reaches the steady motion corresponding to a certain frequency, the next frequency is set, and thus the response curve cannot exhibit the characteristic peaks clearly, i.e., a low resolution is attained. Mitigating the effect of these imbalanced stresses is just a matter of time. Using 397 cm$^3$ samples, the output curves are clear and detailed for sweeping rates below 2.15 Hz/s and the basic pattern of the response curves is not affected by further decreases in the sweeping rate. The same behavior was exhibited by other powders and a sweeping rate of 1.6 Hz/s was chosen for the rest of the experiments.

Figure 3 shows the $F_V$ ~ $f$ curves for various powder samples (constant sweeping rate and acceleration). Each powder has a particular characteristics curve. All the tested powders have common peaks at specific frequencies, and this can there-
fore be considered a feature of any solid particulate material under these measuring conditions, regardless of its composition or physical properties. The vibrational characteristics of the measuring system and the physical properties of the powders intermingle in a complex manner, as reflected in the value of the discrete peaks appearing at given frequencies. At this stage of the investigation, it remains impossible to explain satisfactorily why the peaks and valleys appear at specific vibration frequencies. In spite of this difficulty, it is at least possible to discuss the relative variations of the physical properties of different powder systems by comparing their vibrational characteristics curves.

3. 2 Vibrational characteristics of the powder mixtures

Figure 4 shows the influence of adding small amounts of a second component and the effect that the mixing method has on the beds’ vibrational characteristics. By comparing the $F_v - f$ curve of pure alumina (1) to those of its mixtures (curves 2 ~ 8), it can be seen how the addition of the second component always modifies the output curve, reflecting the adhesion of the minor constituent onto the surface of the alumina particles. The vibrational characteristics of the minor component itself also affects the pattern of the mixtures characteristic curves. Both wax and BN show very low $F_v$ values at almost every frequency, except for those where peaks appear. Therefore, the general tendency of the output curves for the mixtures alumina-wax and alumina-BN must be very similar to that of pure alumina, except at those frequencies where the minor component exhibits peaks. This figure also permits the comparison of the mixture properties as obtained using the two different mixers mentioned above.

Such comparison shows that mixtures prepared by mixer (I) (high speed mixer) have more variable patterns, especially for the alumina-wax mixture (curve 5) in which the coating effect is highly noticeable. For the mixture prepared by adding BN to the alumina-wax mixture, the vertically transmitted force $F_v$ is smaller at every frequency than that of the alumina-wax mixture and, moreover, new peaks appear.

If only BN is added to alumina (4), the general pattern is similar to that obtained with the rotary-type mixer (discussed below); however, some of the peaks below 120 Hz are lost when using mixer (I).

For mixer (II) (rotary-type), although the addition of wax (2) and BN (3) results in variations in the curve, the basic pattern of the pure alumina curve (1) is preserved. The $F_v$ values are globally lower than they are for pure alumina, but the peaks around 140 Hz, which correspond to the pure minor components, remain at the same value.

As mentioned above, both the addition of different components and the differences between the mixers account for the variations in the vibrational response curves.

3. 3 Influence of the concentration of additives

Figure 5 shows the $F_v - f$ curves of alumina-wax mixtures of different concentrations, prepared by the high-speed mixer. All these mixtures were obtained through 90-minutes of mixing under fixed operating conditions. The mixtures reached a temperature of $86^\circ$C and were subsequently cooled to room temperature, after which their vibrational characteristics were measured. Pure alumina was also agitated under the same operating conditions, and although the size distribution and the shape of
the alumina particles before and after agitation were practically the same, the vibration characteristics suffered slight modifications (compare 1 in Fig. 4 — pure alumina before agitation, and Al₂O₃ in Fig. 5 — pure alumina after agitation). At low frequencies (below 80 Hz) \( F_V \) increased for agitated alumina, the peak at approximately 140 Hz was slightly modified, and at frequencies above 330 Hz a new phenomenon appeared — the oscillation of the \( F_V \) value. The differences observed between alumina before and after agitation are due to the modification of the particles' surface characteristics as a result of friction.

As the concentration of wax increases, the peak intensity at 140 Hz decreases; on the contrary, the peak at 60 ~ 70 Hz becomes sharper and more conspicuous. The above-mentioned oscillating phenomenon at high frequencies disappears for wax concentrations above 0.06%. For concentrations of approximately 0.10% the curve pattern clearly changes and preserves its shape through the remaining concentration ranges.

To investigate the extent to which the physical properties of alumina are modified as the wax content increases, we studied the variation in the difference between the transmitted vertical force for mixtures, \( F_V \), and that corresponding to the reference powder (alumina), \( F_{VO} \):
\[ F = F_V(f) - F_V(f_{0}) \]  

Figure 6 shows the relationship between \( F \) and \( f \) for i) alumina-wax mixtures, ii) addition of BN to 0.1% alumina-wax mixture, and iii) alumina-BN mixtures. \( F \) as a function of \( f \) varies depending on the minor component added and its concentration.

Case i) alumina-wax. As the wax concentration increases, the function \( F(f) \) tends toward negative values. The value of \( F \), however, is positive at certain frequencies, which means that the mixture’s packing condition was such that the transmitted vertical force was greater than it was for pure alumina as a result of the reduced friction coefficient.

Case ii). The addition of BN to alumina-wax mixtures results in a further decrease in the value of \( F \), especially at frequencies below 100 Hz.

Case iii). In this case, the 0.02% alumina-BN mixtures obtained by the two mixers are compared. For mixer II (rotary-type), the \( F_V \) curve of the mixture is slightly lower than for pure alumina; however, the pattern is almost the same. The patterns are clearly different when the mixture is prepared with a high-speed mixer. It is thus obvious that for a given powder system the type of mixer used exerts a strong effect on the mixing state (coating) of the resulting mixture. In other words, the state of adhesion of the BN particles onto the surface of the alumina particles depend on the shearing forces acting upon the powder.

3. 4 Influence of the mixing method on the variation ratio \( \eta \)

Next, we evaluated the relative variation \( \eta \) of the vertically transmitted force for those peaks (with large variation \( \Delta F \)) as a function of the minor component concentration. The variation ratio is defined as

\[ \eta = \frac{F_V}{F_{V0}} - 1 = \frac{\Delta F}{F_{V0}} \]  

The relationship between \( \eta \) and wax concentration is shown in Fig. 7. The \( \eta \) ratio decreases as the concentration of wax increases and approaches a stable value at higher concentrations. This suggests that fine particles of wax progressively cover the surface of the alumina particles, and when the degree of coverage is high enough or complete, \( \eta \) attains a stable value. This fact is even more clear when the variation ratio \( \eta \) is evaluated for the peaks appearing at \( 140 \sim 150 \) Hz, which seem to be more sensitive to variations in the physical properties of this particular powder system. For comparative purposes, the sliding friction coefficients of the alumina-wax mixtures were measured by the tilting plate method. It was found that the coefficient values were very close to each other (0.76 ± 0.03) and demonstrated no clear tendencies. When BN is added to wax-coated alumina, \( \eta \) also decreases with increasing concentrations of BN. Finally, when only BN is added to alumina, the tendency is similar, and the \( \eta \) values are close to those obtained for alumina-wax mixtures.

Figure 8 shows the effect that the type of mixer used has on the \( \eta - C \) relationship for alumina-BN mixtures and uses the peaks at \( 140 \sim 150 \) Hz to evaluate \( \eta \). For concentrations larger than 0.004%. \( \eta \) decreases with increasing concentrations of BN. When mixer II (rotary-type) is used, \( \eta \) attains a minimum value at a BN concentration of 0.02% and then suddenly increases. Again, it is assumed that at this point the alumina surface has been completely covered by the fine BN particles. For higher concentrations, the excess fine particles do not adhere to the coarser particles, but
rather remain free, thus modifying the packing state of the powder bed. On the contrary, no minimum value for \( \eta \) is observed when mixer I (high-speed) is used. In this case, the shearing conditions created within the mixer permit further adhesion of the excess fine particles onto the coarser material. In fact, a sample with a concentration of 0.05% was withdrawn and studied, and it was found that there were practically no free BN particles, which means that almost all of them had adhered to alumina particles.

4. Conclusions

An apparatus was tested for detecting the force imparted by a vibrating powder bed onto a sensing sphere immersed in the bed, sweeping continuously at a certain range of vibration frequencies. The evaluation of this force for different samples permitted the detection of the relative variation of the physical properties of the powders.

The pattern of the curve that relates the vertically transmitted force to the vibration frequency depends on the type of powder and the mixing mechanism. The characteristics of the measuring apparatus itself also influence the pattern of the output curve. It was shown that variations in the physical characteristics of the powder can be followed up by studying the peaks appearing at specific frequencies.

By comparing the vibrational response curves of the reference powder (alumina) to those of the mixtures of the reference powder and a minor component (wax and BN), it was shown that the measuring system was highly sensitive to changes in the curves' pattern, and new peaks appeared as a result of the presence of the minor component.

The method also permits the detection of differences existing in the mixing state (the coating of coarse alumina by fine particles of wax and/or BN) as a result of the type of mixer used.

This paper has reported on a comparative study on the peak intensities of a number of mixtures. The investigation is now being focused on how the characteristics curves are related to the physical properties of the powder.

Nomenclature

\[
\begin{align*}
C & : \text{concentration of fine powder [wt\%]} \\
f & : \text{frequency [Hz]} \\
F_V & : \text{vertically transmitted force for the testing powder [N]} \\
F_{V0} & : \text{vertical transmitted force for the reference powder [N]} \\
N_R & : \text{rotary speed of vessel or impeller [s}^{-1}] \\
N_V & : \text{rocking speed of mixer [s}^{-1}] \\
\Delta F & : \text{difference of the transmitted force [N]} \\
\eta & : \text{ratio of the force defined by Eq. (2) [-]} \\
\end{align*}
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References

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