Attrition testing of granules with a tapping sieve

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Abstract

Granules produced to overcome powder handling difficulties must retain their shape during storage or transport but, on the other hand, be easily broken down during designated process. Commonly, granule properties have been evaluated for several properties such as flowability, bulk density, and so forth; however, shape retention and fragility have not yet been evaluated. This study examines whether fragility can be evaluated with attrition tests. We carried out attrition tests with a tapping device on ferric oxide granules and followed the progression of the attrition with time. The attrition mechanism was expressed with an attrition-rate equation with two constants. We showed that these two constants are useful for characterizing the attrition of the granules used in the experiments. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

When we handle finer particles of powdery materials, users increasingly prefer to use them in agglomerated form as granules to avoid difficulties in handling or processing, for example, scattering of fine powder, segregation in mixing or compaction difficulty in forming process. Such granules are often made from a solution by spray-drying or pyrolysis. The granules must have certain properties according to the purpose of their use, but methods currently available for the evaluation of these properties are inadequate because they are mostly the same methods used for powders. For example, if the granules break when discharged from a container, they are not useful. Similarly, in the case of the production of a green body from granules in ceramic field, the granules must break down easily into their primary particles. Furthermore, the granules must be strong enough to resist breakage during classification process.

Thus, granules must have the property of maintaining their integrity during handling, but, on the other hand, they need to possess the appropriate level of fragility or friability so that subsequently they break down easily to their primary particles. An optimum method to evaluate these characteristics is needed. Strength evaluation is often carried out by means of a compression test on a single granule or on a packed granular layer. These tests, however, relate only to the static strength. In addition, compression testing on single granules is troublesome, because many granules must be tested. When we consider fragility, it is useful to introduce the concept of attrition [1]. Attrition refers to any unwanted breakage of particles in any process or handling. An evaluation method that quantifies attrition does not yield a value for strength directly, but it has been recognized as a new method for evaluation of fragility or friability [2].

Existing evaluation methods for attrition can broadly be divided into two main groups: flowing-state tests and static-state tests. Among flowing-state tests are fluidized-bed methods [3–11], pneumatic conveying [12,13], and rotary-drum tests [12,14]; among static-state tests are shear-test methods [15–20]. Fluidized-bed methods usually employ air jets from an orifice smaller than 1 mm in diameter, and they are often applied to catalysts [4], natural raw materials [5], aluminum oxides [6,7], zeolite [8], or other materials [9]. Although particles are often broken...
Fig. 1. Size distributions of ferric oxide granule samples. The initial letter, A or C, in the sample designations stands for the furnace number; the second letter, P or U, indicates whether the sample was purified or unpurified, respectively. Purify means removing silicon and some other impurities. The numeral 1 or 2, if present, stands for type of a spray nozzle.

Fig. 2. SEM photograph of ferric oxide granules: a CP, b AU1.

that the attrition occurred throughout the granule layer rather than at the sieve surface.

2. Experimental methods

Test samples were four kinds of ferric oxide granules produced by pyrolysis of an iron chloride solution in a roaster [24]. Each as-received sample was previously divided into small samples for tests with a chute riffler to avoid variations in quality among the samples. The size distribution of each as-received sample ranged from tens to hundreds of micrometers (Fig. 1). The true density of ferric oxides reported in the literature [25] is 5.2 g/cm³, and the average bulk density of the sample granules measured with a cylinder and a balance was about 0.3 g/cm³. The surface of each granule was like a skeleton with many pores, as shown in the SEM photographs (Fig. 2). As-received granules contained some fragments already attrited. Therefore, we sieved only larger granules from the as-received samples for the attrition tests. To prepare test
Fig. 3. Schematic diagram of the tapping apparatus for attrition testing using test sieves.

Sample, as-received granules were hand-sieved with a 180-μm sieve, taking care that attrition did not occur during sieving.

A cam-type tapping device with a tapping height of 18 mm and a tapping rate of 60 rpm was used for the experiment; a schematic diagram is shown in Fig. 3. The attrition test sieve had a 150-μm aperture just under the 180-μm sieve in the series of the test sieves, a frame diameter of 75 mm, and a sieving depth of 20 mm; it was made of stainless steel or brass, as specified in JIS Z 8801. A sieve of 90 μm in aperture size and a sieve receiver were nested under the test sieve to collect the broken and attrited fragments, respectively. By the adoption of a sieve of 150 μm, whole test samples prepared for attrition tests were regarded as fresh ones with no attrition. The SEM photographs also confirmed that the sieved samples did not contain the attrited fragments. The mass that passed through the 150-μm sieve during the attrition test was defined as the attrited mass.

At the beginning of each test, an initial mass \( m_0 \) of sample was put into the top of the nested sieves. After tapping, the residual mass \( m \) left in the 150-μm sieve was measured to obtain the change in attrited mass \( (m_0 - m) \).

3. Experimental results and discussion

The attrited mass \( (m_0 - m) \) increased with tapping time \( t \), but the rate gradually decreased with \( t \), as in the examples shown in Fig. 4. In all samples, when the initial test-sample mass \( m_0 \) was increased, the attrited mass \( (m_0 - m) \) increased proportionally (Fig. 5). These results show that the effect of the initial mass \( m_0 \) can be disregarded by using the attrition ratio \( 1 - m^* = (m_0 - m)/m_0 \), where \( m^* = m/m_0 \). The relationships between the tapping time \( t \) and the attrition ratio for all test samples are shown in the experimental plots in Fig. 6.

3.1. Attrition mechanism

We proposed two possible mechanisms causing attrition of the granules during sieving: the interaction between the granules themselves, and the contact of the granules against the sieve surface. In the first case, the attrition zone is in the layer of granules, and in the second case, it is on the sieve surface, as shown in Fig. 7.

Which zone contributed most to the observed attrition? If attrition on the sieve surface was dominant, since the mass of the granules in contact with the sieve surface was constant, regardless of the initial mass \( m_0 \), then the attrited mass \( (m_0 - m) \) should also be constant and the attrition...
The attrition rate, defined as the change in the attrited mass with time, decreased with the passage of attrition time (see Fig. 4). The relationship between the attrition rate \((-\Delta m^*/\Delta t)\) and \(m^*\) was investigated. Results are shown in Fig. 8. The attrition rate decreased almost linearly with the decrease in \(m^*\), even when \(m_0\) was changed; the line does not pass through the origin. The intercept \(m^*_c\) on the \(m^*\) axis is defined as the final residue, in other words, the quantity that had not been attrited at the end of the test.

Therefore, the equation for the attrition rate is expressed as follows:

\[
-\frac{d m^*}{dt} = k(m^* - m^*_c),
\]

where \(k\) is a proportionality factor defined as the attrition rate constant.
A simple time-dependent formulation proposed by Gwyn [26] is often cited, but that formulation cannot explain our experimental results, as our attrition rate changed with time. The attrition ratio \(1 - m^*\) can be obtained by integrating Eq. (1). The solution is given subject to the initial condition as follows:

\[
1 - m^* = (1 - m_0^*) \left(1 - \exp(-kt)\right). \tag{2}
\]

The curves predicted by Eq. (2) for each experimental data set are shown in Fig. 6. It can be said that these curves are in good agreement with the experimental results as a whole; they deviate only a little from the experimental values when the tapping time was close to 300 min. The reason for this deviation may be that the granule surfaces changed during tapping for a long time as shown in Fig. 9.

The attrition rate constant \(k\) and the final residue \(m_0^*\) obtained by curve fitting based on Eq. (2) are shown in Fig. 10. \(k\) has a tendency decreasing with increasing \(m_0^*\). The purified granules, indicated by P in Fig. 10, had large attrition rate constants and smaller final residues, while the unpurified granules, indicated by U, had smaller attrition rate constants and larger final residues. Thus, the attrition rate constant \(k\) and the final residue \(m_0^*\) are useful for evaluating the characteristics of attrition. Since the attrition rate constant \(k\) showed some scatter in a range of the large final residue \(m_0^*\) in Fig. 10, further study is needed.

Fig. 9. SEM photographs of ferric oxide granules on the 150- \(\mu\)m sieve after 300 min of tapping: (a) CP (b) AU1.

Fig. 10. Attrition rate constant versus final residue for ferric oxide granules.

Fig. 11. SEM photographs of ferric oxide granules on the 90- \(\mu\)m sieve after 300 min of sieving: (a) CP (b) AU1.
While the attrited purified granules (CP) sampled on the 90 – μm aperture sieve had smooth surfaces as shown in Fig. 11a, some holes were observed on the surfaces of the similarly sieved unpurified granules (AU1) as shown in Fig. 11b. Comparing these observations with the relationship between attrition rate constant and final residue Fig. 10, we conjecture that the unpurified granules are harder to attrite than the purified granules.

4. Conclusions

An attrition-test method using a tapping device and a sieve as the test vessel was proposed as an easy method for evaluating the fragility of granules, especially under conditions of impulsive compression. The attrition tests were carried out with test samples consisting of ferric oxide granules produced by spray pyrolysis of an iron chloride solution. From a consideration of the attrition mechanism, we reached the following conclusions:

1. The mechanism of attrition of granules using this test method was mainly the interaction of the granules themselves in the whole granule layer; attrition by contact of the granules with the sieve surface was negligible.
2. The effect of the sieve material was negligible and can be disregarded in this test method.
3. The attrition rate was well represented by Eq. (1).
4. The fragility of granules was evaluated by the attrition rate constant $k$ and the final residue $m^*_f$ in Eq. (2).

List of symbols

- $k$: attrition rate constant [min$^{-1}$]
- $m$: residual mass on a sieve [g]
- $m_c$: mass of final residue of granules on a sieve [g]
- $m_0$: mass of initial test sample on a sieve [g]
- $m^*$: $m/m_0$ [-]
- $t$: tapping time [min]

References