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An attrition test with a sieve shaker for evaluating granule strength

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Abstract

The purpose of this study is to establish a method for evaluation of the strength of fragile granules such as ferric oxide granules produced by spray pyrolysis from an acid solution of a metallic salt. To examine if they have enough strength to maintain their shape throughout the handling process, and at the same time, enough weakness to break down easily during a specifically designated processing. An attrition test using a Ro-Tap sieve shaker, which is usually used for particle size analysis, was employed. An attrition rate equation was derived from the attrition mechanism of granules on a sieve; a method for the evaluation of strength based on the attrition equation is presented.

Keywords: Attrition; Sieve shaker; Granule; Ferric oxide granule

1. Introduction

Fine powders of ores, foods, medicines, ceramics, and other materials are often agglomerated in various industries and used as granules to overcome difficulties in handling or processing; in addition, solutes are often crystallized from solutions as granules by means of spray pyrolysis. These granules are required to have various properties, depending on the type of granules; above all, granule size and strength are important properties for all types of granules. Granule size must make handling easy, and granules must be strong enough to maintain their shape during handling or processing, yet must easily break down during a specially designated processing. Granule size can be evaluated with sieves or other devices, but an easy and optimum evaluation method for granule strength has not been established. A compression test on a single granule is often carried out for strength evaluation, but this test is troublesome because many granules must be tested. The attrition test, in which granule breakage conditions are similar to those in various processing, would be a practical test for granule strength.

Previously, we [1] proposed an attrition test method using a tapping device, an apparatus for the measurement of tap density, with a test sieve instead of a tapping vessel. In a general attrition test method using a sieve, the granules in the test space may not be affected by the attrited fine particles because they pass through the sieve and are removed; therefore, attrition condition on the sieve would be constant during the test. The use of sieves and sieve shakers in attrition testing is convenient since these devices are commonly used for size distribution analysis.

Bemrose and Bridgwater [2] reviewed attrition tests that used a sieving machine. As discussed in the review, Ho and Hersey [3] expressed a friability index as the inverse of the half-life of the percentage weight remaining on the sieve when 10 g of material was sieved with seven plastic balls of 20 mm in diameter. Rieshel [4] carried out an attrition test by sieving sodium chloride and three kinds of powders with steel balls and compared the attrition rate (expressed as percentage of material passing through the assessment sieve) with that of Ho and Hersey. From these studies, it was found that the assessment results changed depending on the size and quantity of balls for enhancing sieving. Although in these studies the specimens and apparatus used in these studies are not described in detail, such sieving enhancement methods would not be desirable because the enhancing balls complicate the attrition phenomenon and also they add a heavy load for the sieve.

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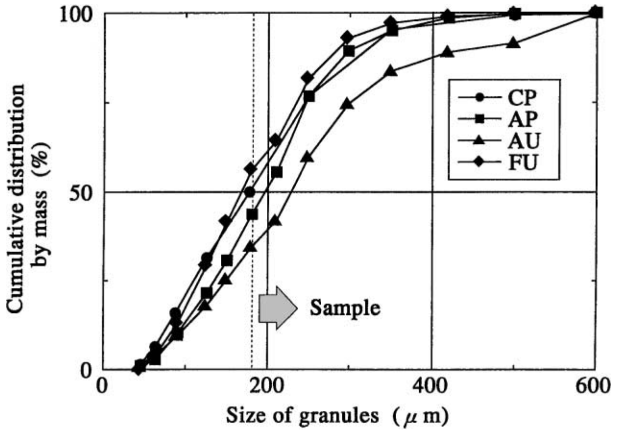

Fig. 1. Size distributions of ferric oxide granule samples. The initial letter, A, C, or F, in the sample designations stands for the furnace number; the second letter, P or U, indicates whether the sample was purified or unpurified, respectively. Purify means removing silicon and some other impurities.

Recently, Bortzmeyer and Goimard [5] compared the attrition rates in a vibrating sieve, without any enhancing ball, for three kinds of fragments of $CaCO_3$ agglomerates of 2–4 mm in diameter that were broken by roll press granulation from samples obtained by compaction. They showed that the evolution with time of the mass of fine particles (under 200 μm) produced during the attrition test obeyed the Johnson–Mehl equation, which is usually used to describe the growth process of recrystallization in metallic glasses; however, the analyses in this study were not based on the attrition mechanism, and the physical meaning of the results was not presented. Bortzmeyer and Goimard also investigated the relation between attrition rates and mechanical properties (tensile strength, compression strength, critical stress intensity factor, fracture energy, fatigue behavior); no correlation was observed.

The purpose of this study is to establish a method for evaluation of the strength of fragile granules. A Ro-Tap sieve shaker usually used for particle size analysis was used for the attrition test, and an attrition rate equation was derived from the attrition mechanism of granules on a sieve. A method for the evaluation of strength using the attrition equation is presented, and the physical meanings of the attrition evaluation are made clear.

2. Experimental method

Test samples used were four kinds of ferric oxide granules produced by spray pyrolysis of an iron chloride solution that was used for rinsing steel plates, similar to samples used in previous study [1]. Each as-received sample had been previously divided into small samples for tests, using a chute riffler to avoid variation in quality among the samples. The size distributions of each as-received sample ranged from tens to hundreds of micrometers (Fig. 1). Average bulk density using a measuring cylinder and a balance was about 0.3 g/cm^3; the density reported in the literature [6] is 5.2 g/cm^3. The surface of the granules consists of many pores (Fig. 2). As-received samples were hand-sieved with a 180-μm sieve, taking care that attrition did not occur during sieving, and the fine

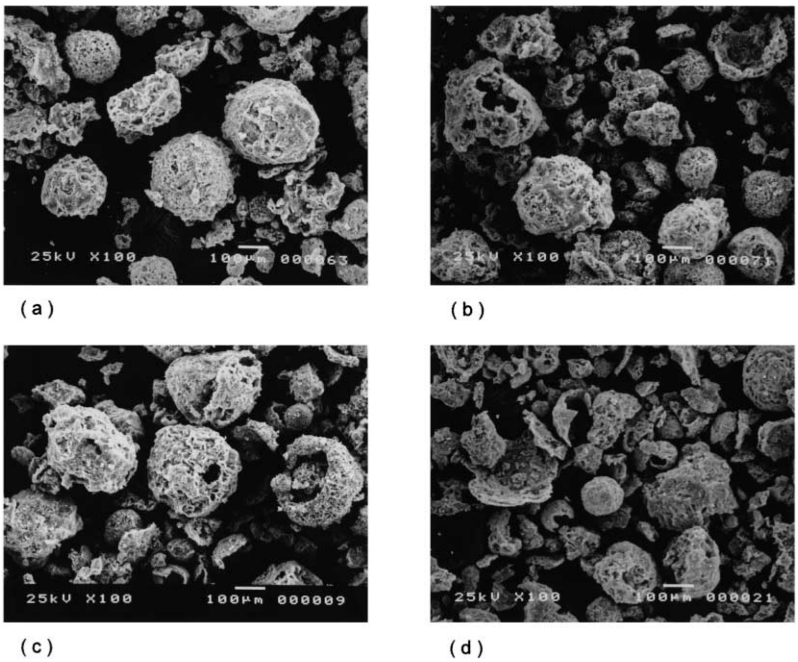

Fig. 2. SEM photographs of ferric oxide granules: (a) CP, (b) AP, (c) AU, and (d) FU.

particles, which included any fragments already attrited, were removed.

The sieve shaker used in the attrition tests was a Ro-Tap type sieve shaker with a rotating rate of 290 rpm, stroke of 25 mm, and tapping rate of 165 min^{-1} (IIDA SIEVE SHAKER by Iida Seisakusho). Aperture size of test sieve (manufactured by stainless steel) for attrition was 150 μm, frame diameter was 200 mm, and sieve depth was 20 mm, as specified in JIS Z 8801. It is conceivable that fine particles smaller than 180 μm were present as a residue in the test samples, even with use of the 180-μm sieve. Therefore, if a sieve of 180 μm was used for the attrition test, particles finer than 180 μm would pass through the sieve at the beginning of the test and would be mistaken as granules attrited during the attrition test. Thus, by adoption of 150 μm for the attrition test, whole test samples were regarded as non-attrited before the attrition test began. In practice, when we put the test samples into the sieve, no granules passed through. A sieve of 90 μm in aperture size to collect the broken fragments and a sieve receiver were nested under the sieve. The quantity of material that passed through the 150-μm sieve during the attrition test was defined as the attrited quantity.

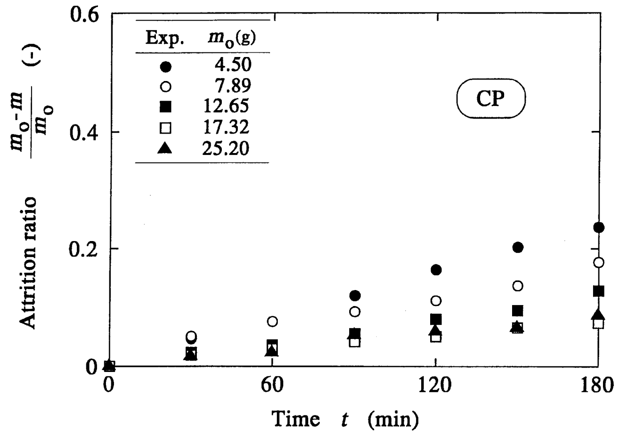

Fig. 4. Relationship between attrition ratio to mass of the initial test sample and attrition time.

3. Experimental results and discussion

3.1. Experimental results

The initial mass m_0 was placed in the sieve; attrited mass $(m_0 - m)$ was determined by measuring the residual mass m on the 150-μm sieve after operating the sieve shaker for sieving time t. The results (Fig. 3) show that the attrited mass $(m_0 - m)$ increased linearly with time t and that the attrition rate can be regarded as constant. Also, the attrited mass $(m_0 - m)$ increased with increasing initial mass m_0. However, when the attrition ratio was considered, the attrition rate was higher with a smaller initial mass m_0 and appears to approach a constant value with increasing initial mass m_0 (Fig. 4). Here the attrition ratio and the attrition rate are defined as $(m_0 - m)/m_0$ and $(1/m_0)(\mathrm{d}m/\mathrm{d}t)$, respectively. The results in shown in Figs. 3 and 4 are different from those of the tapping method reported previously [1], in which the attrition rate decreased with attrition time and the attrition ratio was constant, regardless of the initial mass m_0; this discrepancy may be due to differences in the attrition mechanism.

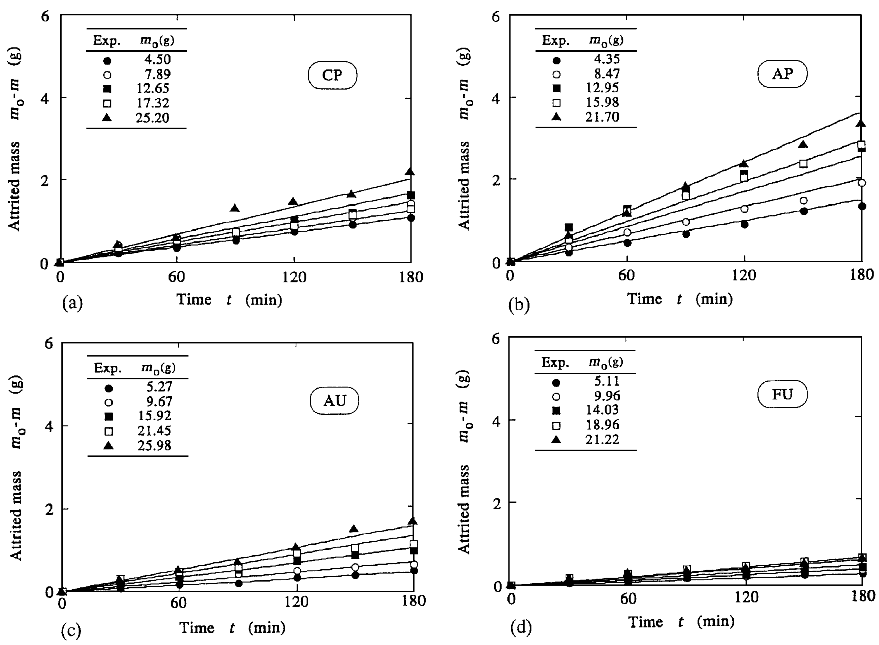

Fig. 3. Experimental data for (a) CP, (b) AP, (c) AU, and (d) FU with results (solid lines) drawn according to Eq. (5).

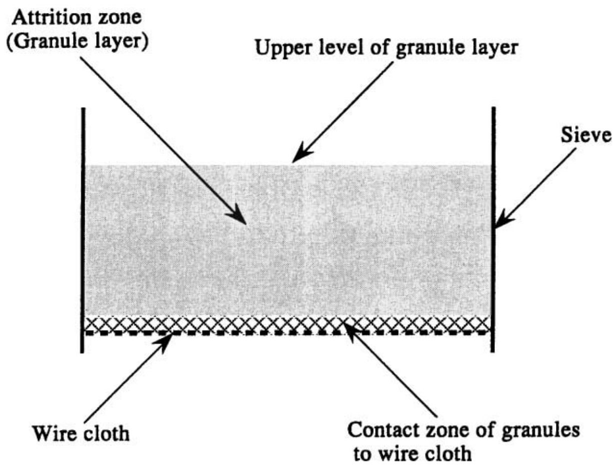

Fig. 5. Explanation of the attrition zone in the granular layer.

3.2. Attrition mechanism and attrition rate

To analyze the mechanism of attrition, we can separate attrition behavior on a sieve into two zones (Fig. 5): a contact action zone, where granules are in contact with the sieve surface, and an interaction zone consisting of granules in the bulk granule layer.

When attrition occurs by contact action of granules with the sieve surface, the attrition zone is always constant; therefore, the attrition rate is constant regardless of the quantity on the sieve, as long as the granules cover the full surface of the sieve.

On the other hand, when attrition occurs in the granule layer, the attrition ratio should be constant in the whole zone, regardless of the granule quantity on the sieve. As a result, the attrition rate decreases in proportion to the granule quantities on the sieve, as the granule quantities in the layer decrease with progression of the attrition.

Thus, when we consider that attrition using a sieve occurs in both granule zones, the attrition rate equation can be described as

$$-\frac{\mathrm{d}m}{\mathrm{d}t} = k_1 + k_2 m \tag{1}$$

where m is the granule quantity on the sieve, t is attrition time, and k_1 and k_2 are attrition rate constants for the contact action of granules at the sieve surface and the interaction of granules in the bulk granule layer, respectively. Integration of Eq. (1) from $t=0$ (at this time, $m=m_0$) to $t=t$ gives the attrition quantity

$$m_0 - m = \frac{1}{k_2}\{1-\exp(-k_2 t)\}(k_1 + k_2 m_0). \tag{2}$$

This equation is basic to attrition using a sieve. It demonstrates a convex dependence of the attrition quantity $(m_0 - m)$ on attrition time t and shows that attrition quantity $(m_0 - m)$ increases with initial mass m_0.

3.3. Attrition with the Ro-Tap type sieve shaker

The experimental results for the Ro-Tap sieve shaker indicate a linear dependence of the attrited mass $(m_0 - m)$ on time t. If in Eq. (2),

$$k_2 t \ll 1 \tag{3}$$

then, at short attrition times,

$$\exp(-k_2 t) \doteqdot 1 - k_2 t \tag{4}$$

and the attrition rate will become linear.

To determine if this approach was correct, an attrition experiment was carried out using a receiver only, without a sieve; results with and without a sieve are compared in Fig. 6. The attrited mass $(m_0 - m)$ obtained with the attrition test using only the receiver is extremely small, even if both the interaction attrition of the granules themselves in the granule layer and the contact attrition of the granules to the bottom plate of the receiver are contained in the result. From this result, it can be concluded that the attrition rate in the granule layer is very slow and that the attrition rate constant k_2 is very small.

Therefore, by substituting Eq. (4) for Eq. (2), we obtain

$$m_0 - m = (k_1 + k_2 m_0)t. \tag{5}$$

In this equation, the attrited mass $(m_0 - m)$ increases in proportion to the time t and the initial mass m_0, which is similar to the experimental results.

Now, if k_R (attrition rate by experiments) represents the slope of the plot of attrited mass $(m_0 - m)$ vs. time t in the experimental results, k_R has the following relation with Eq. (5),

$$k_R = (k_1 + k_2 m_0). \tag{6}$$

k_R can be obtained as the slope of a linear line by the method of least squares. Although the behavior of k_R is different for each granule sample, in all cases, the relation between k_R and m_0 is linear (Fig. 7). Since the figures of

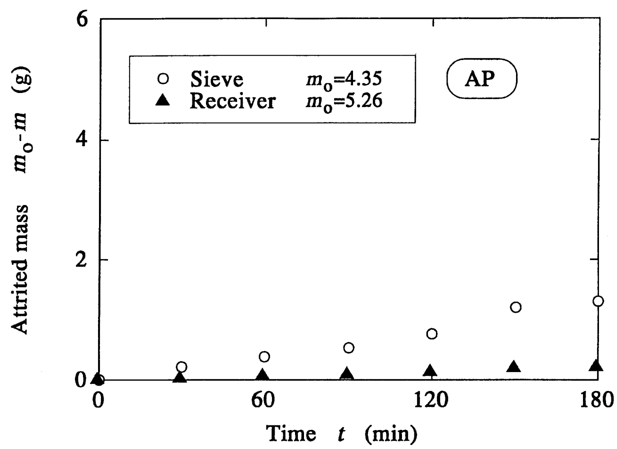

Fig. 6. Comparison between masses attrited on test sieve and on receiver only.

the values of t and k_2 obtained from Fig. 7 are 2 and -4, respectively, as shown in Fig. 10, then the value of $k_2 t$ is much smaller than 1. Therefore, the validity of the assumption of Eq. (3) is confirmed. Accordingly, the calculated results of Eq. (5) using the values of k_1 and k_2 obtained from Fig. 7 coincide with the experimental for all samples, as shown by the solid lines in Fig. 3.

Consequently, it is clear that the experimental results of the attrition test using the Ro-Tap type sieve shaker and a test sieve for ferric oxide granules are well described by Eq. (5).

3.4. Meaning of the attrition rate constant k_1

We assumed that the attrition of ferric oxide granules by the Ro-Tap sieve shaker occurs almost completely from contact action of granules with the sieve surface (attrition rate constant k_1), because attrition from interaction among the granules themselves was very small (Fig. 6). This contact action consists of two factors: wear of granules from the rotary motion of the sieve and impact breakage due to vibration of the sieve surface by the tapping hammer. To investigate which factor was dominant, an attrition test using rotation of the sieve only, without hammer tapping, was carried out; attrition that occurred during this test was due only to wear.

Comparison of the results of attrition tests with and without hammer tapping show that attrition from wear in the Ro-Tap sieve shaker was small (Fig. 8). Accordingly, in the attrition test using the Ro-Tap sieve shaker, it can be presumed that the vibrating strength of the sieve due to impact by hammer tapping is important. To confirm this presumption, an attrition test was carried out using a Ro-Tap sieve shaker with the hammer covered with a gum plate, which results in a smaller impact force. Attrition progression using the hammer covered with gum was slower than that with the usual steel hammer (Fig. 9). Accordingly, it is clear that attrition occurred almost totally from the impact force of the hammer. The attrition rate constant k_1, therefore, refers to granule fragility arising from vibration of the sieve due to the impact force of the hammer.

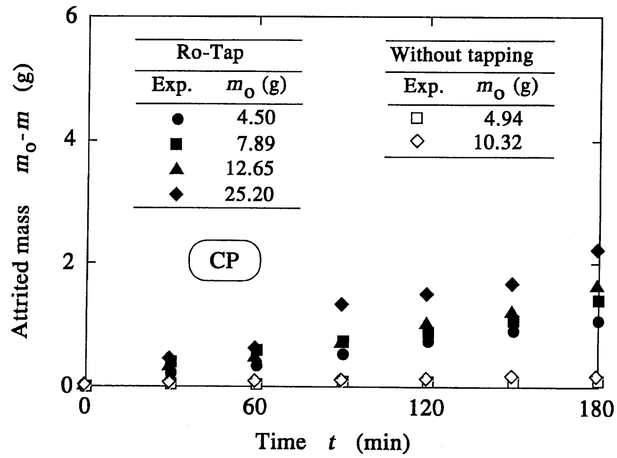

Fig. 8. Comparison between masses attrited with the Ro-Tap shaker with and without tapping.

3.5. Differences in attrition in the granule layer between tapping and Ro-Tap sieve shaker methods

In the tapping method [1], attrition in the granule layer was dominant, but this type of attrition was small with the Ro-Tap sieve shaker method. We will now consider why the contribution to attrition in the granule layer is different in the two methods, in spite of the same interactions there. We presumed that in the tapping method, attrition occurs from the impact force of compression, based on the inertia of the granules themselves, as the whole granule layer falls at each tapping action. On the other hand, in a Ro-Tap sieve shaker, the impact force of the tapping hammer is transferred only from the sieve surface to the granules; the granules are bounced on the vibrating sieve surface and

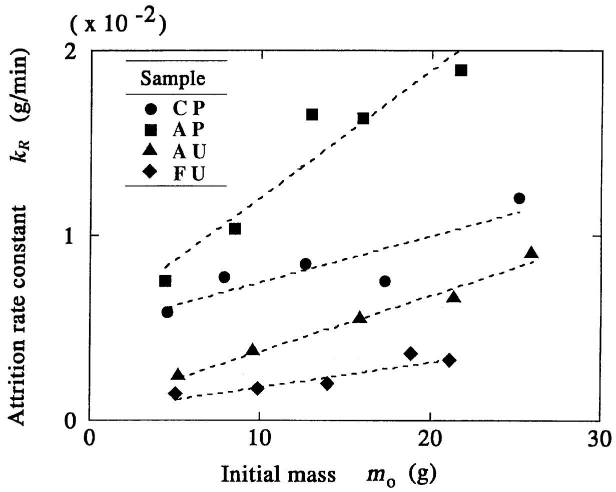

Fig. 7. Relationship between initial mass and attrition rate constant.

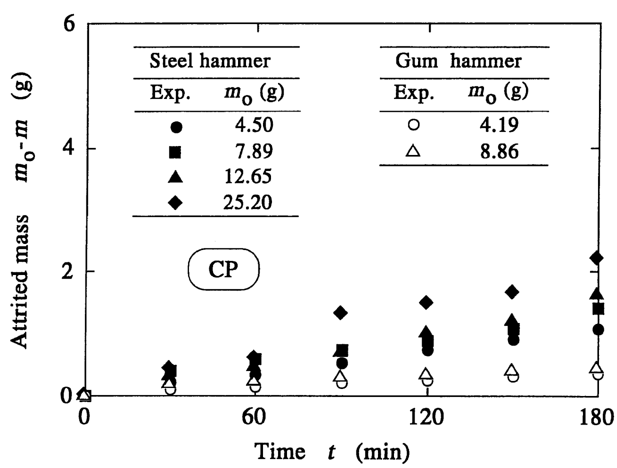

Fig. 9. Comparison between masses attrited with the Ro-Tap shaker with the usual steel hammer and with the hammer covered by gum.

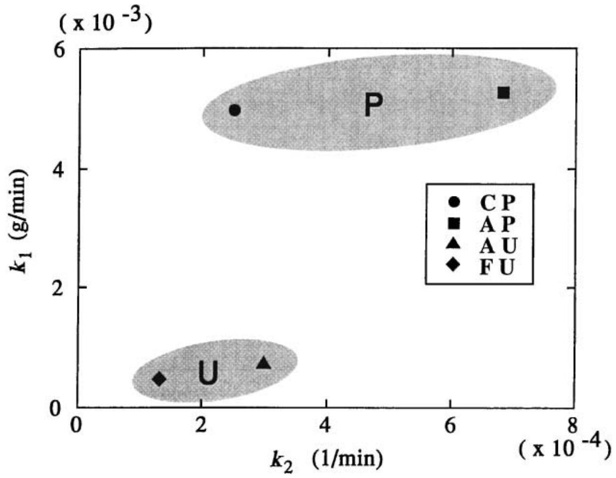

Fig. 10. Characteristic attrition values for ferric oxide granules obtained with the Ro-Tap sieve shaker method.

dispersed into the space on the sieve, but not compressed. Accordingly, we concluded that in attrition with the tapping method, impact breakage by compression is dominant, whereas in the Ro-Tap sieve shaker, wear based on the interaction of the granules themselves is dominant, even if attrition occurs in the granule layer; therefore, the attrition mechanism changes, depending on the type of force acting on the granules.

3.6. Evaluation of granules based on results of attrition tests

Attrition characteristics of samples can be distinguished by construction of an attrition evaluation figure (k_1 vs. k_2; Fig. 10). In these samples, k_1 is greater for the P granules (purified ferric oxide) than for the U granules (unpurified), indicating that breakage of the P granules is easier than breakage of the U granules. Also, k_2 of the AP granules is greater than that of the CP granules, indicating that, of the P-type granules, the AP granules wear more easily. Consequently, in the attrition test method using the Ro-Tap sieve shaker, the physical meaning of the attrition rate constants k_1 and k_2 are clear, in contrast to those measured with the tapping method. Therefore, the fragilities for impact breakage and wear can be simultaneously evaluated with the Ro-Tap sieve shaker attrition test.

4. Conclusion

We have studied methods to evaluate the strength of fragile granules by an attrition test using a sieve. The attrition rate in the sieve was considered in two attrition zones with different attrition mechanisms; in this study, we assumed that the attrition rate was due to the sum of the attrition mechanism in each zone. Based on this assumption, the attrition rate equation was derived. The attrition equation applied to the Ro-Tap sieve shaker was compared with experimental results for ferric oxide granules. The following conclusions were obtained.

(1) The attrition rate in an attrition test using a sieve can be represented by Eq. (1) as a summation of rates from the attrition mechanisms of two action zones: a contact action zone, where granules are in contact with the sieve surface, and an interaction zone consisting of granules in the bulk granule layer. The attrition quantity was obtained (Eq. (2)) as a resolution of the rate equation.

(2) In the attrition quantity obtained using the Ro-Tap sieve shaker (Eq. (5)), the contact action of granules with the sieve surface is dominant.

(3) In the attrition test using a sieve, the characteristics of breakage by contact action of the granules to the sieve surface and wear by the interaction of granules themselves in the bulk granule layer are represented by k_1 and k_2 (Eq. (1)), respectively.

We expect that our conclusions also pertain to sieve shakers other than the Ro-Tap sieve shaker used and that this test method will result in further practical test methods by providing the capability to select and standardize appropriate sieve and impact forces for materials.

List of symbols

k	attrition rate constant of tapping method (1/min)
k_1	characteristic constant (g/min)
k_2	characteristic constant (1/min)
k_R	attrition rate coefficient by experiment (g/min)
m	residue on a sieve of 150 μm (g)
m_0	initial mass on a sieve of 150 μm (g)
t	time (min)

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