Small Scale Tests for Attrition Resistance of Solids in Slurry Systems

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In many present day solids-liquid contacting processes and slurry transport systems, the attrition rate of the solid particles is of primary technical and economic concern. Particle size degradation in a transport pipeline may make the delivered solids entirely unsatisfactory for the intended use, or separation of fines from the liquid phase and makeup of attrited solids may be so costly that an otherwise attractive process becomes unacceptable.

Pilot scale tests to measure attrition in a slurry system are often costly and very time consuming. For this reason small scale tests have been developed for screening candidate systems prior to pragmatic pipe-loop or pilot-plant tests. Two of these test methods, crushing tests on individual particles, and annulus flow in a rotating cup apparatus, appear to give useful data on the relative attrition resistance of various solids and to provide a basis for estimating the attrition rate to be expected in a flowing system.

Results obtained by using these test methods with silica gel in hexane and with molecular sieve particles in kerosene yielded some information as to the predominant mechanisms of attrition for these two types of solids. The brittle, glasslike silica gel appears to fail through impact fracture of small to medium size chips from the large particles, while attrition of molecular sieves results from surface abrasion, probably due to failure of the binder material, and yields only extremely fine products of attrition.

This paper, which is based on a rather limited amount of preliminary experimentation, has been prepared in the hope that it may stimulate additional work on the development of useful standardized procedures for evaluating attrition resistance of solids.

While a great deal of work has been done in the study of slurry flows since 1906, when two of the earliest papers on the subject were published (*1*, *2*), most of these investigations have been concerned only with measuring pressure drop and minimum velocity for solids transport, and have not considered attrition of the solids. Only a few comparatively recent studies deal specifically with particle attrition or make significant mention of it. Some of these pertain to gas-solids systems (*3*, *4*) where conditions are quite different from those typical of slurry flows, and thus the test results can not be applied to solids-liquid systems. Other papers briefly mention attrition experienced in solids-liquid systems, but only in a very qualitative way (*5* to *9*). Thus, there exists neither quantitative data on the attrition rate of solids in slurry systems, nor accepted or standardized laboratory tests for measuring attrition resistance. The present work describes two test techniques which have been used in the study of particle attrition and presents some attrition data for two solids-liquid systems.

The simpler of the two tests involves measuring the energy required to crush individual particles. Comparison of the fracture energy distributions for various solids indicates their relative strengths and, within certain limitations, attrition resistance.

The other test more closely simulates a practical slurry system. A cylindrical shell rotating within a concentric annular flow passage causes continuous circulation of a suspension of solids around the annulus. Measurement of the size distribution of the solids before and after extended periods of operation gives the attrition rate in the test system. The attrition rate in a full scale slurry system can be estimated by extrapolation.

INDIVIDUAL PARTICLE CRUSHING TEST

In this test, discrete particles are mechanically crushed in a device which simultaneously measures the force applied to the particle and the deformation it undergoes. From these data, the energy required to produce fracture is calculated.

Description of Apparatus and Test Procedure

This apparatus is built around a rigid frame having a platform which can be raised or lowered smoothly by means of a micrometer screw and handwheel. Mounted from the top of the frame is a metal bob with a small blunt tip. A strain-gage force transducer measures the force applied to the bob by the platform, and this signal is recorded on one channel of a dual channel recorder. The displacement of the platform is measured by a magnetic motion transmitter whose output is recorded on the other channel of the recorder.

In the performance of a test, the particles to be crushed are scattered over the metal bottom of a small pan having transparent plastic sides. The pan is filled with whatever liquid is of interest for the solids-liquid slurry, and is then placed between the movable platform of the test apparatus and the bob. With the aid of a binocular microscope sighted through its transparent sides, the pan is positioned so that a single particle is directly below the bob. The recorder is started and the platform gradually raised until the particle is crushed between the bob and the pan. When fracture is observed through the microscope, the platform is lowered and the pan repositioned with a different particle under the bob. For each sample of material to be tested, approximately 30 to 40 particles are crushed, and the distribution of fracture energies is calculated and plotted.

Test Results and Evaluation

Particle crushing tests were performed on several different solids which are of interest in slurry contacting systems. These included silica gel, ion exchange resins, urea and molecular sieves. Only some typical test results for silica gel and for molecular sieves will be presented here as examples.

Figure 1 shows fracture energy distribution for samples of silica gel in hexane. The data for fresh slurry and for the sample that had been repeatedly heated and cooled are essentially the same; this indicates that heating and cooling had no significant effect on particle strength. In contrast, there is a marked decrease in particle strength due to sudden exposure to water and the liberation of the

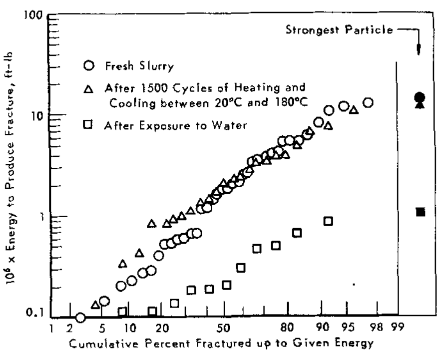

Fig. 1. Individual particle crushing test results: fracture energy distribution for 300 to 400 μ silica gel particles in hexane.

high heat of adsorption of water on silica gel. Over the entire distribution, the energy required to produce fracture in this sample was lower by almost an order of magnitude than in the fresh slurry.

For all of the samples of silica gel crushed, except the one exposed to water, it was observed that the least energy which resulted in fracture was about 10^{-7} ft. lb. In a flow system one might therefore expect that if the kinetic energy of a silica gel particle were less than 10^{-7} ft. lb., then collisions with pipe wall or other stationary objects would not result in particle fracture. For a 350μ silica gel particle, with hexane filling its pores, this kinetic energy corresponds to a velocity of about 10 ft./sec.

Figure 2 shows the crushing strength data obtained from samples of molecular sieve particles. There is no large, clear-cut, strength advantage for either of the materials over the entire distribution, although sample *B* appears to be stronger at both ends of the distribution.

All of these molecular sieve materials appear to have greater mechanical strength than the silica gel particles tested. However, the use of these data directly to predict the relative attrition resistance of silica gel and molecular sieves, without consideration of the physical differences between the two materials, would lead to an erroneous conclusion. Data presented in a subsequent section of this paper indicate that silica gel is far more attrition resistant than the apparently stronger molecular sieve material. An explanation for these results can be given in terms of observed fracture characteristics of the two materials. For the very angular, brittle silica gel particles, fracture in the crushing test consists of just the breaking off of a small chip, and the resulting particles are similar in appearance to those observed after actual slurry-flow attrition. The molecular sieve material, on the other hand, is rounded and rather plastic, the force applied by the crushing device is distributed over a large area, noticeable distortion of the particle is observed before fracture, and fracture consists of breaking the particle into several approximately equal fragments. These fragments are quite different from the extremely fine particles shown to result from actual slurry-flow attrition. Thus the mechanical crushing test is not a good analog of the attrition process for materials like molecular sieves. Direct application of crushing test results to the prediction of attrition resistance should be restricted to brittle, glassy materials like silica gel where impact fracture rather than abrasion predominates.

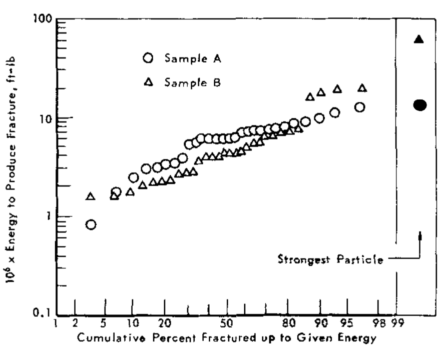

Fig. 2. Individual particle crushing test results: fracture energy distribution for 300 to 420 μ molecular sieve particles in kerosene.

ANNULUS FLOW TEST

In this test, a slurry is caused to flow continuously around an annular passage where the particles experience repeated collisions with the wall and each other. The resulting attrition is determined from periodic analysis of the particle size distribution of the solids.

Description of Apparatus and Test Procedure

A drawing of the annulus flow test apparatus giving its pertinent dimensions is presented in Figure 3. The apparatus is built from three pieces of different diameter plastic tubing. The smallest cylinder is closed by a disk cemented inside its top end, and the largest and smallest diameter tubes are cemented concentrically to a plastic base to form an annular passage ¾ in. wide and 6½ in. deep. The intermediate size tube is cemented at the top to a plastic disk which in turn is connected to the rotating shaft of a small motor. The body of the motor is fastened to another plastic disk which serves as a cover for the annular tank and also positions the rotating inverted cup (formed by the intermediate size tube and its end disk) concentric to the other cylinders. Where the flammability of the slurry being tested requires it, an air motor is used to drive the rotating cup. Otherwise, a small variable speed electric motor, which provides more reliable speed control, is used. Speed of rotation is determined stroboscopically.

As the starting material for an attrition test in this apparatus, a sample of solids having a known size distribution is prepared by sieving, elutriation, or other convenient means. A quantity of solids having a settled volume of about 100 ml. and a total slurry volume of about 800 ml. is most suitable. With this amount of solids uniformily distributed over the bottom of the tank, the inverted cup can be lowered into position without contacting and crushing the particles. (Larger quantities of solids may be used if rotation of the cup is started and the solids are suspended before the cup is lowered into its final position.) The rotation of the cup causes the liquid and the

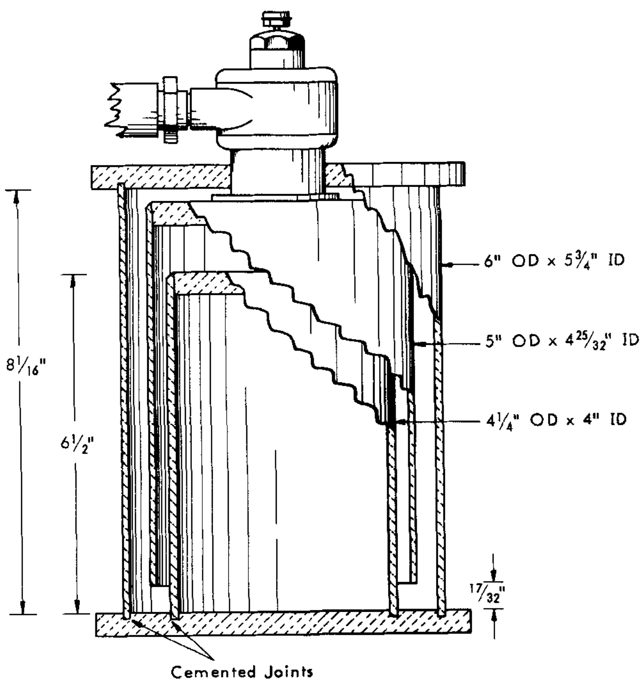

Fig. 3. Schematic diagram of annulus flow test apparatus.

particles to flow continuously around the annulus and the particles to experience repeated collisions with the walls and each other. The flow patterns which result from rotation of the cup within the annulus are somewhat more complex than those which would be encountered in flow through a straight pipe. For all practical operating conditions, under which the annulus flow test apparatus might be used, the region between the rotating cup and the outer wall is in the flow regime where cellular Taylor vortices prevail (*10*). This flow pattern was clearly observable during test operation. The region between the rotating cup and the inner wall would be expected to have only simple circular flow, although it is possible that some disturbance might be communicated from the outer area through the space below the bottom edge of the inverted cup.

During the time required for measurable attrition, the speed of rotation is periodically checked, and for volatile liquids, evaporation losses are made up with fresh liquid. At the end of the test, the solids are removed from the apparatus, and the new particle size distribution determined. The same solids sample is used for several successive tests if it is desired to investigate the progressive attrition over a long period. The entire solids sample may be returned to the apparatus, or the fines produced by attrition may be excluded from further tests.

Test Results and Evaluation

Annulus flow attrition tests have been performed using two industrially interesting slurries, silica gel in hexane and molecular sieves in kerosene. Samples of silica gel in hexane were tested at several rotor speeds; except for one preliminary period of operation, molecular sieve material was tested at only one speed because of its low resistance to attrition.

In the tests with silica gel in hexane a quantity of solids having about 170 ml. settled volume was used. The initial size distribution contained only material which passed a 35 mesh screen and remained on a 48 mesh screen (295 to 420μ). Since most actual slurry processes can tolerate some size reduction of the initial particles, the products of attrition for these tests were arbitrarily taken to include only those particles that passed through a 100 mesh screen (147μ) after the tests.

Tests with silica gel in hexane were run at rotor peripheral velocity of approximately 10 and 14 ft./sec. Additional testing at 6 ft./sec. was attempted, but the operation of the air motor drive at this low speed was very unstable. Electric motor drive was not used because of the flammability of the slurry, and a speed reducer was not available for the air motor. No data are reported at this low speed because they were very erratic and unreliable.

Data from the tests with silica gel in hexane are presented in Table 1, and the results of two of the tests at 10 and 14 ft./sec. are plotted on Figure *4a*. The curves are seen to start off with a relatively steep slope (high rate of attrition) which subsequently decreases to a moderate value. It is believed that the high initial attrition is due to the presence of flawed particles in the samples at the start of the tests. Such particles have been observed in microscopic examination of fresh gel. They look cloudy (as though they contained many internal cracks or flaws) rather than glossy and transparent as do most of the gel particles. Such particles would be expected to break easily during the first part of the test, and with their removal the attrition rate should decrease toward a steady value. Even in the absence of these severely flawed particles, one must expect a range of particle strength (as indicated by the crushing tests previously described) due to slight flaws or only the variation in particle shape. As attrition proceeds, the stronger members of the original population remain, and the rate of attrition decreases.

The curve representing a test velocity of 14 ft./sec. seems to have reached a relatively constant slope representing a rate of attrition of about 0.0018%/hr. The final slope of the curve for 10 ft./sec. corresponds to about 0.0005%/hr., but appears to be still decreasing. As an attempt to determine a steady state attrition rate for this test condition it was decided to repeat the 10 ft./sec. test, carrying it out to a greater time of operation. The results of the two tests at 10 ft./sec. are shown on Figure *4b*. The attrition rate during the last interval of the 500-hr. test is 0.00011%/hr., and appears to be somewhat less

TABLE 1. ATTRITION OF SILICA GEL IN HEXANE (To Size Smaller Than 147μ)

Time	Attrition rate during interval %/hr.	Particle size distribution cumulative % finer than 147μ	295μ	420μ
Test run at 14 ft./sec.				
start of test		0	0	100
	0.01079			
after 18 hr.-45 min.		0.202	2.908	100
	0.00270			
85 hr.		0.381	4.529	100
	0.00184			
150 hr.		0.501	4.171	100
test run at 10 ft./sec.				
start of test		0	0	100
	0.00654			
after 16 hr.		0.105	8.649	100
	0.00242			
83 hr.-20 min.		0.268	7.863	100
	0.00077			
148 hr.-25 min.		0.318	11.207	100
Repeat				
Test run at 10 ft./sec.				
start of test		0	0	100
	0.00123			
after 17 hr.		0.021	2.143	100
	0.00051			
96 hr.		0.062	2.665	100
	0.00030			
192 hr.		0.092	3.081	100
	0.00020			
336 hr.		0.120	2.883	100
	0.00011			
505 hr.		0.139	2.831	100

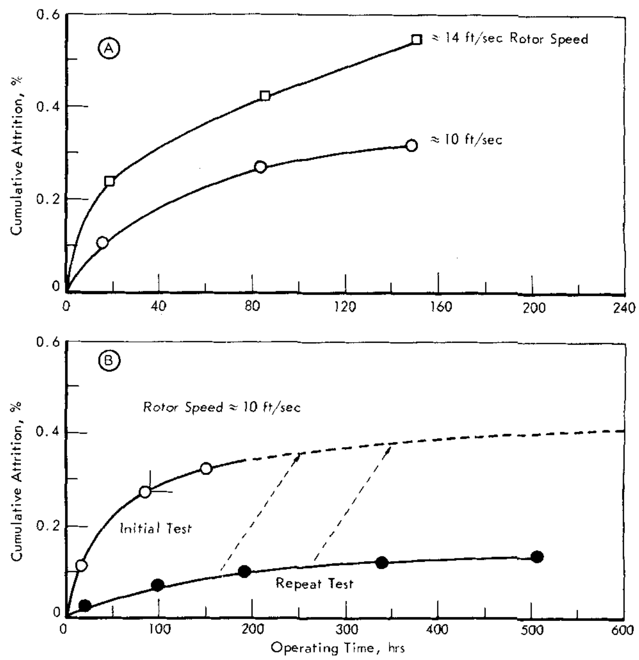

Fig. 4. Annulus flow test results: attrition of silica gel in hexane.

than the preceding interval. However, at this low attrition rate, uncertainty in particle size measurement becomes important, and the difference may not be significant.

In a previous section it was observed that the particle crushing tests on silica gel indicated that 10 ft./sec. might be a threshold velocity below which attrition would be slight. The annulus flow tests seem to verify this value. The 40% increase in velocity from 10 to 14 ft./sec. produced an order of magnitude increase in attrition rate, which is such a strong dependence on velocity that the lower value, 10 ft./sec., might essentially be considered a threshold.

An unexpected aspect of the 500 hr. test may be seen from the two curves on Figure 4*b*. Although both tests were carried out using nominally the same type of silica gel, and were performed using identical experimental techniques, the observed attrition rate at the start of the 500 hr. test is remarkably lower than that for the 150 hr. test. This might lead one to question the reliability or reproducibility of the test method, yet within each test the measured attrition rates during different time intervals are remarkably consistent. One must conclude that the tests are reliable and that there is an actual difference between the two materials tested. The 500 hr. test was carried out at a considerably later date, and the material for the two tests did not come from the same shipment of gel. Apparently, the material used in the 500 hr. test contained fewer weak or cracked particles and thus did not undergo the initial high attrition rate which is believed to result from such particles.

If one translates the curve for the 500-hr. test to such a position that a part of it coincides with the curve for the 150-hr. test, an interesting conjecture may be made. (The 500 hr. curve is shown in this translated position on Figure 4*b*.) Although the evidence would be more conclusive if the length of the overlapping portion of the curves were greater, it appears that the material used in the 500 hr. test behaves the same as the other material after about 90 hr. of operation that is, as though it had been preattrited by this period of operation. Starting from this new base point, the two materials appear to behave similarly. Thus, in a plant operation, while one might find different initial rates of attrition upon adding different charges of gel to the system (depending upon the production history of the material), one might expect that eventually the same low rate would be reached. The difference in total attrition for various charges might be expected to be a fraction of a percent of the initial charge and would have only minor influence on the economics and operation of the process.

Particle size distribution measurement of the silica gel samples after test operation, and microscopic examination of the fines, showed that a full range of sizes from about 1μ up to the initial large particles was present, and that even the very fine particles had the usual glassy, angular appearance characteristic of larger silica gel particles. The mechanism of attrition in this brittle, glassy material is apparently high impact fracture of small to medium size chips from the larger particles.

Several grades of molecular sieve material were tested. To prepare the samples, the molecular sieve particles were first shaken in a mixture of kerosene and *n*-tetradecane so that the sieves could adsorb the normal paraffin. The liquid was then decanted and the particles washed repeatedly with isopentane so that when they were dried they would be free flowing and could be sieved to determine particle size. The oversized and undersized particles were discarded to provide an initial size distribution covering a limited size range.

A preliminary test with molecular sieves was carried out at a rotor speed of 10.7 ft./sec. After only a short period of operation it became obvious that the attrition rate was extremely high and that this was not a realistic operating condition for this particular slurry; the actual tests with molecular sieve material were carried out at a speed of 5.3 ft./sec. (With this less flammable liquid, an electric motor drive with better speed regulation permitted low speed operation.) Several samples of different materials were prepared so as to have essentially identical initial size distribution. A few drops of Shell AntiStatic Additive, ASA-3, were added to the kerosene to reduce the tendency toward agglomeration during the tests. The test with each material covered 83½ hr. of operation, with the particle size distribution also measured after the first 16½ hr. of operation.

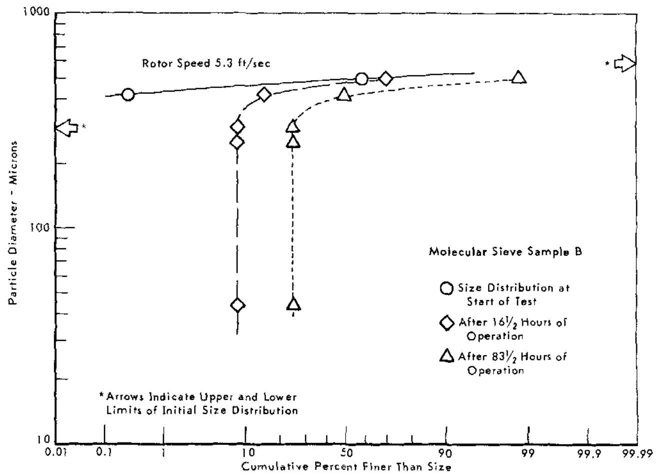

Fig. 5. Annulus flow test results: change in particle size distribution due to attrition.

The initial, intermediate, and final particle size distributions measured in a typical test are shown on Figure 5. Test results for several samples are presented in Table 2.

Samples *A* and *C* had very low attrition during the first 16½ hr. of operation, but only Sample *C* maintained a low rate for the entire test period. Sample *A* apparently has a hardened surface which resists initial attrition, but when this surface has been worn through, the core attrites quite rapidly. Sample *C* has only about 3%w. binder material rather than the more common 20% and appears to be harder throughout.

Study of the particle size distributions and microscopic examination of the particles after the tests gave some valuable information as to the probable mechanism of attrition for the molecular sieve materials. The size distributions of the materials after the tests show that in no case was there any significant amount of material in the 44 to 295μ range (indicated by the nearly vertical lines between these sizes on Figure 5). Microscopic examination of the material finer than 44μ showed it to consist almost entirely of particles of about 1μ diam., or of loose agglomerates of these particles. These ultimate particles could barely be resolved by the optical microscope. The material coarser than 295μ had essentially the same appearance as before exposure to attrition (irregularly shaped rounded particles with no sharp corners or fracture surfaces).

The conclusion drawn from this evidence is that attrition of these molecular sieve particles occurs not as an impact fragmentation of the large particles into several of various smaller sizes, but instead as abrasion of extremely fine particles from the surface of the large ones. Only in this way can the absence of intermediate size particles and the observed shape of the large particles after attrition be explained. Such a mechanism is also very reasonable

TABLE 2. ATTRITION OF MOLECULAR SIEVES IN KEROSENE
(To Size Smaller Than 250μ)
All Tests Run at 5 ft./sec.

Time	Attrition rate during interval %/hr.	Particle size distribution cumulative % finer than 44μ	250μ	295μ	417μ	495μ	589μ
Sample "A"							
start of test		0	0	0	0.256	57.322	100
	0.0403						
after 16½ hr.		not measured	0.665	0.672	3.042	63.160	100
	0.2737						
83½ hr.		18.818	19.005	19.033	37.075	94.508	100
Sample "B"							
start of test		0	0	0	0.271	57.324	100
	0.5006						
after 16½ hr.		8.222	8.260	8.265	14.881	69.285	100
	0.2481						
83½ hr.		24.692	24.884	24.914	48.455	98.587	100
Sample "C"							
start of test		0	0	0	0.272	57.336	100
	0.0563						
after 16½ hr.		0.911	0.929	0.940	3.220	59.171	100
	0.0119						
83½ hr.		1.697	1.726	1.757	5.169	62.445	100

when it is realized that the large molecular sieve particles are actually agglomerates of very fine particles held together by a binder. Attrition probably results from a failure of the mechanical bond between the binder and the fine particles which make up the molecular sieve particles.

Since surface abrasion is apparently responsible for attrition of this type of material, then the roughness of the surface against which the particles rub must be important, and considerably worse attrition could be expected in a relatively rough metal piping system than in the smooth plastic test apparatus.

DISCUSSION AND SUMMARY

Two small scale attrition tests that might be developed into standardized test methods have been described. The annulus flow test appears to be generally suitable for evaluating the attrition resistance of particles. Since it actually involves flow of a slurry, it can be made to simulate most nearly the attrition conditions to be encountered in a practical installation.

The individual particle crushing test which is much simpler and faster, is most applicable to brittle angular particles such as silica gel, which seem to be attrited as a result of high-impact fracture. However, it may have some usefulness when comparing other types of material, if the materials being considered are similar to one another (for example, different grades of molecular sieves). For very disimilar materials the mechanical strength of the particles as determined by this test does not necessarily correlate with attrition resistance because different attrition mechanisms may predominate for the different materials. For example, molecular sieve particles show greater strength in the crushing test than do silica gel particles, while in the annulus flow attrition test it is found that the molecular sieves are attrited much more rapidly. The explanation for this seeming anomaly is that attrition of molecular sieves actually results from surface abrasion rather than from higher energy fragmentation of the entire particle, as occurs in the crushing test. Attrition of silica gel, on the other hand, results from the fracture of chips from the large particles, and this same phenomenon is observed in the crushing test.

From the annulus flow test one obtains attrition rate data which should be almost directly applicable to a plant system. One factor which introduces uncertainty in the prediction of full-scale attrition rates from the annulus flow data is the difference in surface to volume ratio for the two cases. If the relative importance of particle-wall collisions and particle-particle collisions were known, then this difference could be accounted for. Tests in annulus flow devices of several sizes would provide this information.

Another source of uncertainty in extrapolating from the annulus flow data to a plant attrition rate is the lack of flow similarity between a piping system with straight runs, elbows, valves, etc., and the annular test device. If the velocities throughout the plant systems are comparatively uniform, this probably introduces only moderate error. If, however, there are points in the plant flow system where the particles are briefly exposed to a very high fluid velocity (for example, in the educator of a liquid lift system) then there may be a considerable degree of uncertainty. Data from the annulus flow test, which is carried out at constant speed, are not obviously applicable to a situation in which the flow is rapidly accelerated and decelerated.

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Manuscript received February 10, 1967; revision received October 11, 1967; paper accepted October 12, 1967.