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STUDIES ON GRANULATION IN FLUIDIZED BED I.

METHODS FOR TESTING THE PHYSICAL PROPERTIES OF GRANULATES

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The knowledge of the properties of granulates is of considerable importance,both as regards application and scientific research work. Methods for the determination of the most important physical characteristics of granulates (such as grain-size distribution,density characteristics, pore space fraction, rolling tendency characteristics, and mechanical strength) published in literature, are described. The results of the research work aimed at checking the applicability of the testing methods and the development of new techniques are reported. The concepts of "loosened bulk density" and "rolling tendency coefficient" are introduced and measuring techniques enabling the determination of these quantities are described. A simple measuring technique is proposed for the determination of the pore space fraction. The technique is based on the identical space filling characteristics of particles of an identical shape. A new measuring method for the determination of the abrasion strength (abrasion resistance) of the granulates is described, which is based on the measurement of the mechanical stress acting in the fluidized layer.

In chemical and related industries (e.g. the food industry) the granulates prepared are used partly as starting materials of further products and partly as final products. Various fields of

application raise various requirements concerning the quality of the granulates. However, the basic principles of qualitative characterization are nearly identical and mainly refer to the physical properties of the granulates.

The main physical characteristics of granulates are the following:

a) grain size distribution and form of the grains,
b) density characteristics,
c) porosity,
d) rolling tendency characteristics,
e) mechanical strength.

Naturally, in addition to the above-mentioned physical parameters, there are other characteristics which may be of importance in some fields of application, e.g. tendency to crumbling, and pressing ability, etc. However, the knowledge of these - although in some cases of considerable importance - is in general not as essential as the physical parameters listed earlier. Several other authors are of the same opinion [1, 2, 3, 4, 5,etc.].The granulates are in some cases, mainly in the pharmaceutical industry, very often qualified in addition to their physical properties on the basis of indirect parameters, such as the physical characteristics of the tablets made of them (e.g. variations in the weight of the tablets, time of falling apart, and abrasion resistance) [6, 7].

In the following, testing methods found in literature for the determination of the most important physical characteristics of granulates will be described, together with a report on the elaboration of new techniques and the improvement of known ones.

GRAIN-SIZE DISTRIBUTION AND FORM OF THE GRAINS

In the study of the granulation process, generally the distribution of heaps of grains of different properties according to

size is to be determined. The grain-size distribution of the starting material to be granulated can rarely be determined by sieve analysis; sliming [8, 9, 10], sedimentation [8, 9, 10] or microscopic examination [3, 9, 11] can be applied instead. The latter method also enables the form of the grains to be observed.

The most widely used method for the determination of the grain-size distribution of granulates is sieve analysis. This test is most frequently carried out by a standard set of sieves [18, 9, 10]. The granulates - especially those obtained by building-up type granulation - are of nearly spherical form, or at least the largest and smallest dimensions of the granulate are not significantly different from each other and consequently the accuracy of the method is satisfactory.

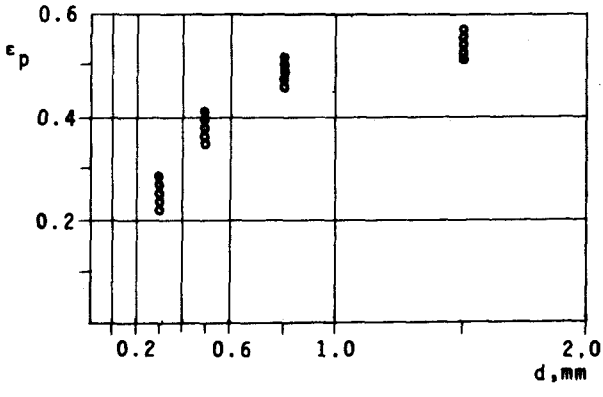

Fig. 1

In the case of granulates prepared by the fluidization granulation technique it was found that the porosity of the granulates increases, i.e. the grain density decreases with increasing dimensions.

Fig. 1 shows the change in pore space fraction plotted against increasing volume in the case of 5 different granulates, prepared from a sand fraction of 0.1 to 0.2 millimetre grain size in a laboratory-scale fluidization granulation apparatus. It can be concluded from the Figure that the grain density of granulate particles exceeding the 0.6 millimetre size is decreased to about one half as compared to that of the ungranulated 0.1 to 0.2 millimetre fraction.

In the opinion of the author, in those cases where - the porosity of the granulates and, together with it -, the grain den-

sity is dependent on grain size, the ratio of the grain spaces is far more characteristic of the granulates than the weight ratio of the individual fractions. The former can be calculated from the weight ratio, if the porosity is known. In this case it is not the weight ratio of the grains, but their volume ratio that is to be considered in the calculation of the average grain size and other average values (e.g. average grain density, etc.) of the granulate.

DENSITY CHARACTERISTICS

In the case of a heap composed of porous grains, real density, grain density and bulk density can be considered.

The Real Density is often difficult to determine, because the simple pyknometer density determination technique [8, 9, 10, 11] does not yield reliable results unless the sample is compact, readily wettable and of small grain size. The Biltz vacuum pyknometer [9] can be used to determine the density of fine powders, if it is possible to find an adequate indifferent measuring liquid. The liquid-medium pyknometer technique can be used for the determination of the real density of masses composed of porous grains only with reservation.

The liquid used for the measurement penetrates the grains to an extent depending on the structure of the grains and the characteristics of the liquid; consequently an intermediate value is obtained which is between the real density and the grain density. In such cases, the Hofsass air-pyknometer can be used; the accuracy of the latter can be increased by using helium instead of air [9].

Grain Density: the mass of grains of unit volume, can be expressed in the following way:

$$\rho_g = \rho(1 - \varepsilon_p) \tag{1}$$

where

ρ_g is the grain density (gram/cu.centimetre),

ρ is the real density (gram/cu.centimetre),

ε_p is the pore space fraction.

The techniques applicable for the determination of the pore space fraction are described in the next chapter.

Bulk Density: the mass of a heap of unit volume of grains. The value of this quantity depends on a number of parameters, such as average grain density, grain-size distribution, and grain form, etc. However, the most influental parameter is - for a given heap of grains - the closeness of packing. Accordingly, three bulk density values can be defined: close-packed bulk density,filled-in bulk density and loosened bulk density.

Close-Packed Bulk Density: the mass of a unit-volume heap of grains pressed together intensively. This value is to some degree dependent on the method of compression [12, 13]. In general, it can be stated that the highest degree of compression and, together with it, the highest close-packed bulk density is attained by vibration brought about in some way, e.g. pneumatically. However, even this value differs only slightly from grain heap densities obtained by some other compression technique, e.g. mechanic or manual compression. Different authors have proposed various techniques for the determination of the close-packed bulk density. For example, NEWITT and CONWAY-JONES [2] used high-frequency vibration, MARKS and SCIARRA [7] repeated manual knocking in a graduated cylinder; according to KONCZ [8], the best method is the application of a Becker-Rosenmüller shaker, etc. Consequently, it is very difficult to compare the measured results. Nevertheless it is a general opinion that - provided proper care is exercised - any of the methods is adequate for the determination of the close-packed bulk density.

Filled-in Bulk Density. The mass of a grain heap, when filled into a vessel of unit volume. In addition to grain size distribution, grain density and grain form, the filled-in bulk density also depends on the size and shape of the volumetric vessel and on the method used to fill in the grains. The values obtained with different techniques differ and this necessitated standardization of the filled-in grain density determination methods [3]. The ap-

paratus consisting of two parts, shown in Fig. 2, serves this purpose [15]. The essence of the measurement is that a 120 millilitre portion of the granulate heap to be tested is poured into the funnel and permitted to freely flow into the graduated cylinder of 100 millilitres capacity. The excess is removed from top of the cylinder and the mass of the latter is weighed. Other authors [9] have proposed the application of a filling apparatus according to Gary and Böhme.

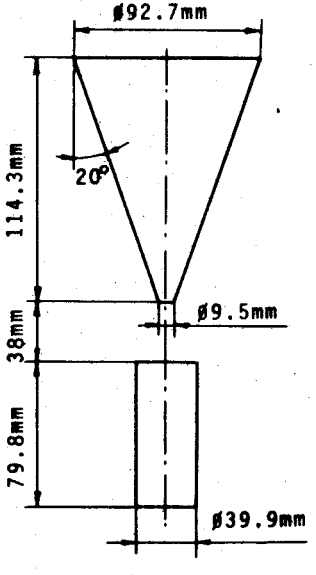

Fig. 2

Loosed Bulk Density: the mass of a unit-volume heap of loosened grains. The loosened heap is produced in the following manner: a granulate heap of known mass is placed into a simple laboratory-scale fluidization apparatus (4-5 centimetres in diameter) and fluidized with air until an expansion of one and a half times or twice the volume of the original, and then the amount of air is decreased until a stationary layer is obtained. The height of the layer is measured and it enables the loosened bulk density to be calculated in a simple manner:

$$\rho_l = \frac{4\,G}{D^2 \pi Y_m} \qquad (2)$$

where

ρ_l is the loosened bulk density (gram/cu.centimetre),

G is the mass of the heap of granulate (gram),

D is the diameter of the apparatus (centimetre),

Y_m is the minimum fluidization layer height (centimetre).

The schematic drawing of the apparatus is shown in Fig. 3. While carrying out the measurement, care should be exercised to ensure that, on the one hand, Y/Y_m should not be higher than 1.5

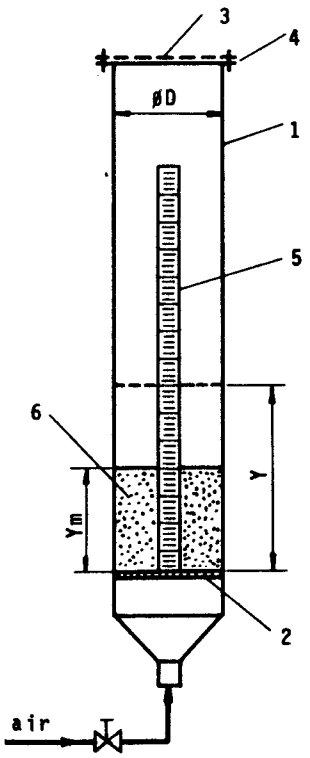

Fig. 3

1. Fluidization apparatus
2. Fritted glass retaining disk
3. Sieve, 15-20 μm
4. Dismountable flange
5. Millimetre scale
6. Loosened grain heap

to 2, in order to avoid the production of flow dust, and, on the other, Y_m/D should be 0.5 to 1.5. The loosened bulk density values determined by the proposed technique are well defined and reproducible, and the apparatus and the procedure are simple. To know the value of the loosened bulk density is important both from the points of view of plant operation and design.

POROSITY

The pore space fraction - which is the ratio volume of the pores present in the grains by total volume of grains - can be determined by a number of techniques. One of these is based on the principle that a non-wetting liquid (mercury) is forced at different pressures into the pores of the grains, the amount of the liquid is measured and thereby it is possible to draw conclusions not only on the pore space fraction, but also on the size of the pores [10, 11]. This measurement, with the use of mercury, can be carried out in an even more simple manner, if it is not necessary to know the distribution of the pore space fraction and the latter can be determined by a suitable pyknometer technique [3, 8, 12].

In the study of the granulation operation it is necessary to determine the pore space fraction on a large number of granulate heaps, moreover, the determinations should be carried out quickly. For this purpose, a simple but adequate pore space fraction determination method was developed in the author's laboratory. The new

method is based on the fact that the void fraction of a relatively narrow grain fraction of practically spherical, compact grains scatters between narrow limits [16]. Any significant deviation from this value is in the case of grains larger than 0.2 millimetre - and, accordingly, generally also in that of the granulates - the consequence of the porosity of the grains and hence it can be used for pore space determination.

The definition of the void fraction is the following:

$$\varepsilon' = \frac{V_r - V}{V_r} = \frac{F \cdot Y - \frac{G}{\rho}}{F \cdot Y} \tag{3}$$

where

ε' is the void fraction,

V_r is the volume of the layer (cu.centimetre),

V is the volume of the solid material present in the layer (cu.centimetre),

G is the mass of the solid material (gram),

ρ is the real density of the solid material (gram/cu.centimetre),

Y is the height of the layer (centimetre),

F is the cross section of the apparatus (sq.centimetre).

On the basis of geometric considerations it is evident that the void fraction, in the case of porous grains, can be written in the following manner:

$$\varepsilon_2' = \varepsilon_1' + (1 - \varepsilon_1') \cdot \varepsilon_p \tag{4}$$

where

ε_2' is the void fraction in the case of porous grains,

ε_1' is the void fraction without pores (taking only the free space between the grains in consideration),

ε_p is the pore space fraction of the grains.

Equation (4) can be written for the point of minimum fluidization:

$$\varepsilon_{m2}' = \varepsilon_{m1}' + (1 - \varepsilon_{m1}') \varepsilon_p \tag{5}$$

and hence the pore space fraction of the grains (ε_p) can be expressed:

$$\varepsilon_p = \frac{\varepsilon'_{m2} - \varepsilon'_{m1}}{1 - \varepsilon'_{m1}} \tag{6}$$

where

ε'_{m1} is the minimum void fraction of a compact granular material of a form similar to that of the porous grains,

ε'_{m2} is the minimum void fraction of the porous granular material.

The minimum void fraction of the porous granular material (ε'_{m2}) is, on the basis of Equation (3), the following:

$$\varepsilon'_{m2} = \frac{Y_m - \frac{G}{F\,\rho}}{Y_m} \tag{7}$$

where

Y_m is the minimum fluidization layer height of the grain fraction (centimetre),

G is the mass of the weighed-in grain fraction (gram),

ρ is the mean real density of the materials building up the grains (gram/cu.centimetre),

F is the cross section of the apparatus (sq.centimetre).

The minimum fluidization layer height of the individual granulate fractions can be determined, as described in the previous section, by means of the simple laboratory fluidization apparatus shown in Fig. 3. The narrower the fraction tested (i.e. the higher the number of fractions into which the heap of granulate was divided), the higher the accuracy of the pore space fraction determination.

During the research work carried out in connection with the layer expansion of fluidized systems, among others the minimum void fraction of relatively narrow grain fractions of quite a number of compact granular materials were determined. Microphotographs of the grains were prepared and compared with those of porous grains and granulates made of various starting materials by different procedures. The minimum void fraction of grain fractions consisting of compact grains of approximately identical form shows a good agreement, and consequently - in the case of grain fractions of a size exceeding 0.2 millimetre - the following values can be substituted into Equation (6):

a) regular, approximately sphere-shaped, porous grains: $\varepsilon_{ml} = 0.45$;

b) less regular, sphere-shaped porous grains, granulates prepared by a rotary (e.g. rotating disk) apparatus: $\varepsilon_{ml} = 0.50$;

c) even less regular, porous grains, granulates prepared by fluidization: $\varepsilon_{ml} = 0.55$;

d) broken, porous grains prepared by crushing or rough disintegration, granulates prepared in a fluidized layer from needle-crystal shaped starting material: $\varepsilon_{ml} = 0.60$.

The determination method can naturally be refined in such a manner that a series of photographs is made of compact grains of various forms, the photograph of the porous grains to be tested is compared with these, and the actual minimum void fraction value is determined by this comparison. However, it is very rarely necessary to carry out this procedure in studies connected with fluidization granulation since the shape of the granulates obtained - except for some needle-crystal shaped starting materials - is approximately identical, and the value of $\varepsilon_{ml} = 0.55$ can be substituted into Equation (6).

Two comparative tests were carried out as regards the applicability of the measuring technique described earlier. The essence

of the first was that the pore space fraction of a number of granulate fractions was determined with the proposed technique, and afterwards three lots of one thousand grains were counted, their mass weighed and the pore space fraction (ε_p^*) calculated by Equation (1). The deviation between the values determined by the two methods plotted against the pore space fraction, as determined on the basis of the minimum void fraction, is shown in Fig. 4. It can be concluded on the basis of the Figure that except for a few cases, the relative deviation is smaller than ± 10 per cent and the agreement is better at medium values, i.e. in the case of granulates of medium size. In the second test, the minimum void fraction of burnt clay grain fractions of known porosity was determined and afterwards the pore space fractions were calculated by Equation (6) in such a way that - based on microscopic comparison - a value of $\varepsilon'_{m1} = 0.50$ was substituted. A comparison of the pore space fractions showed that the relative deviation in this case is also generally below ± 10 per cent.

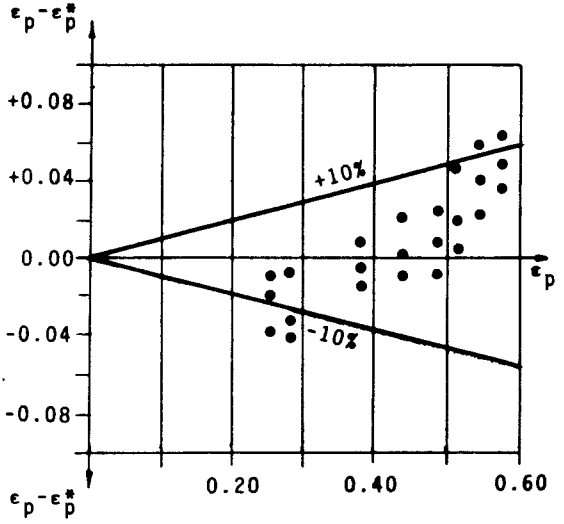

Fig. 4

It is the advantage of the proposed measuring technique that it is simple, rapid, and no expensive equipment is needed.

On the other hand, it is burdened by the drawback that when applying it, the granulate heap to be tested has to be separated to at least 5 to 6 grain fractions. However, this is carried out anyway during the sieve analysis and so the two tests can be connected. The average pore space fraction, characteristic of the

whole heap, can be calculated, knowing the grain size distribution, by weighted averaging.

ROLLING TENDENCY CHARACTERISTICS

Rolling tendency is an important physical property of granular heaps of materials and it is characterized by the internal friction of the heap. The rolling tendency characteristics are generally described by the slump angle of a heap produced in some manner, or by the rate of flow from a funnel of defined dimensions.

In connection with material heaps, a number of angles can be defined,such as slump angle, falling angle, sliding angle, spatula angle, internal friction angle, and inclination angle [17]. Most frequently it is the slump angle from among these, referred to a heap of grains produced by a standardized procedure, which is determined. The essence of one such procedure is that the standardized funnel [15] shown in Fig. 2 is fixed in such a position that its lower end is 4 centimetres above the base and the material to be tested is poured into the funnel until the apex of the material heap produced just reaches the lower end of the funnel. The height of the heap and the diameter of the base circle are measured and the slump angle calculated from these data [3]. Slump angle determination can also be carried out by the Langhaus-apparatus [9].

When a heap of grains flows from a funnel of standardized dimensions, the result can be expressed by the discharge time of the material of the unit mass or unit volume, and by the mass or volume of material discharged during the unit time, etc. For example, GOLD and his co-workers [18] evaluated the results on the basis of the discharge time of a given mass, KANENIWA and IKEKAWA [19] on the basis of the mass discharged during unit time, and LISKE and MÖBUS [20] on the basis of the volume discharged during unit time.

A number of authors dealt with the circumstances of the discharge of granular matter from a funnel, including KANENIWA and IKEKAWA. These authors concluded [19] that, in addition to the properties of the grain heap, the rate of discharge depends, to a large extent, on the diameter of the discharge opening, its length and the cone angle of the funnel, but it is independent of the height of the heap above the opening. The latter statement, with a few exceptions, was also confirmed by other authors [21, 22]. It follows from the aforesaid that it is primarily necessary to standardize the funnel in order to obtain comparable results. For example, the standard funnel shown in Fig. 2 [15] enables the rolling tendency characteristics to be determined. A hundredfold (in grammes) of the density (gram/cubic centimetre) of the solid is poured into the funnel and the time necessary for discharge is measured [3]. Experience shows that the researchers use funnels of different dimensions for this measurement [4, 5, 7, 19, 20].

In the author's opinion, the discharge data are more characteristic of the rolling tendency than the slump angle. This is supported by the fact that the discharge rate shows - in the case of different grain heaps - considerably greater variations, and consequently the method is more sensitive. For example, according to data published by LISKE and MÖBUS [20], the discharge rate from a given funnel corresponding to a slump angle of 46° was 288 millilitres/minute, whereas that corresponding to 44.2° was 370 millilitres/minute. On the other hand, the discharge rate, in the case of certain types of granulate heaps, may fluctuate in time even if the average value is the same [18]. The determination of this fluctuation provides a possibility for further refinements.

Different methods based on the measurement of the discharge rate from a funnel are used in general practice for the determination of the rolling tendency characteristics and consequently it seems desirable to introduce a few improvements in the measuring techniques in order to obtain results that are readily comparable. The grain volume discharged in unit time seems to be more adequate for the comparison of the rolling tendency characteristics of gra-

nulate heaps than the mass of grains discharged in unit time. This is especially true if heaps of different grain density are to be compared. Of course, the average grain density or porosity should be known in this case; a simple and rapid technique for its determination was described in the previous chapter.

The other problem arises as a consequence of the difference of the testing methods. It is the author's opinion that this difficulty could be overcome if the results obtained with different methods were compared with the discharge rate of a generally accepted standard material. The rolling tendency coefficient defined in this manner is

$$\varphi = \frac{v_g}{v_s} \tag{8}$$

where

φ is the rolling tendency coefficient,

v_s is the discharge rate of the standard material (cu.centimetre/second),

v_g is the discharge rate of the granulate under test (cu.centimetre/second).

Narrov fractions of different sizes of a number of different materials were examined and from among the materials available, that consisting of regular glass spheres of approximately 0.15 mm (100 mesh) dimension was found to possess the most advantageous rolling tendency characteristics. This material is a commercial product (GLASS BEADS FOR GAS CHROMATOGRAPHY, approximately 100 MESH, BRITISH DRUG HOUSES LTD., B.D.H. LABORATORY CHEMICALS DIVISION, POOLE, ENGLAND) and consequently it seems to be adequate to be used as a standard in the determination of rolling tendency characteristics.

If in the measurement of discharge rate, amounts corresponding to the same grain space are weighed in from the standard material and from the granulates, and the discharge times of the to-

tal quantities are measured, Equation (8) takes the following form:

$$\varphi = \frac{\tau_s}{\tau_g} \tag{9}$$

where

φ is the rolling tendency coefficient,

τ_s is the discharge time of the standard material (sec),

τ_g is the discharge time of the granulate heap under test (sec).

The time of discharge of a grain space of 100 cubic centimetres (G_s = 296 grammes) of the standard material proposed in the foregoing from the standard funnel shown in Fig. 2 [15] is τ_s = 8 seconds, i.e. v_s = 12.5 cubic centimetres/second. The rolling tendency coefficient of a few grain heaps are given in the following:

glass beads	d = 0.25 millimetre	φ = 0.93
	d = 0.42 millimetre	φ = 0.82
sand	d = 0.10-0.20 millimetre	φ = 0.62
	d = 0.20-0.32 millimetre	φ = 0.68
	d = 0.32-0.40 millimetre	φ = 0.66
	d = 0.40-0.50 millimetre	φ = 0.63
	d = 0.50-0.63 millimetre	φ = 0.62
	d = 0.63-0.80 millimetre	φ = 0.60
	d = 0.80-1.00 millimetre	φ = 0.54

According to the author's experience, the rolling tendency coefficient of granulates prepared by the fluidization process is in the 0.3 to 0.6 range. Probably there exists some material whose discharge rate is greater than that of the standard material proposed, but the rolling tendency coefficient of the overwhelming majority of the materials is between 0 and 1.

MECHANICAL STRENGTH

Mechanical strength means the totality of those properties which express the resistance of the granulates against mechanical stresses [1]. The mechanical stress may be pressure, impingement, and abrasion, etc. Often these actions simultaneously manifest themselves during the operations carried out with the granulates, such as shipment, storage, feeding, and packing. The methods used for the measurement of the mechanical strength of granulates can be classified into two groups. The methods belonging to one group enable the compressive strength to be determined, and the other the abrasive strength.

The underlying principle of the methods used for the determination of the compressive strength of granulates is the following. A compressive stress is put on an individual granulate grain and the force is increased until the grain is crushed. The compression strength is most frequently expressed as the ratio maximum compressive force before crushing per cross sectional area of the grain [1, 2, 4, 23]. The drawback of this method is that it does not adequately model the mechanical stresses acting upon the granulates in their use.

Abrasion resistance of the granulates means their resistance against abrasion effects encountered during their application [5, 7, 24]. Abrasion resistance is most frequently determined by a sieve analysis carried out after the abrasive action and the result is presented as the ratio fraction remaining on a sieve of a given mesh size to the total quantity of sample weighed in. The methods used to produce abrasive mechanical stress are multifarious. MARKS and SCIARRA [7] applied the Roche pulverization tendency testing apparatus [25]. FUNNER and his co-workers [26] shook the granulates in a closed container for a given period of time and afterwards sieved them. DAVIES and his co-workers [5] modified the standard testing procedure developed for the testing of coal [27] and abraded the granulates in a rotary shaking-mixing apparatus. A standard [24] recommends that as long as there is no ade-

quate standard for an abrasion testing apparatus, it is preferable that the interested parties should come to an agreement as to the conditions of the test.

In connection with studies on fluidization granulation, the question arose as to the method to be applied for the determination of the abrasion resistance of granulates. Either the application of some sort of shaking apparatus could be taken into consideration, or abrasion in a fluidized layer could be used. In order to settle this question, a series of experiments were carried out with a granulate heap prepared in a fluidized layer of a sand fraction of 0.1-0.2 millimetre size with gelatine as the binding agent. The experiments were carried out in such a manner that a given quantity of the granulate heap to be tested was exposed to different abrasive stresses for a given period of time. Thereupon the grain size distribution was determined by sieve analysis. The abrasive mechanical stress was brought about in the following manners:

a) A 100 gram portion of the granulate was placed into the receiver of a set of sieves, covered with the lid, and was shaken with a horizontal motion at a frequency of 200/minute on top of a "Labor MIM" shaking apparatus for 10 minutes (R 1),

b) The experiment was repeated with the above-described parameters in such a manner that 25 steel balls of 8 millimetre diameter were placed into the vessel together with the granulate sample (R 2),

c) A 100 gram portion of the granulate was weighed into a 300-millilitre Erlenmeyer flask, the latter was fixed into one of the clamps on the side of a "Labor MIM" shaking apparatus and shaken at a 200/minute frequency for 10 minutes (R 3),

d) A 100 gram portion of the granulate was kept in a fluidized state in a laboratory fluidization apparatus of 5 centimetre diameter (cf. Fig. 3) at a threefold layer expansion (Y/Y_m = = 3) with an air stream for 10 minutes (F 1).

The results of the experiments are summarized in Table 1. X is the ratio average grain diameter of the grain heap after abrasion by average grain diameter of the grain heap to be tested (percentage).

Table 1

	Granulate to be tested	Granulate after abrasion			
		R 1	R 2	R 3	F 1
Average grain diameter (millimetre)	0.55	0.52	0.27	0.38	0.41
X, per cent	100	94	49	69	74

It is apparent from the Table that the horizontal shaking of the granulate is not sufficiently effective (R 1). If there are also steel balls in the vessel when carrying out horizontal shaking, the abrasive stress is too strong (R 2). Shaking in the Erlenmeyer flask (R 3) and abrasion by fluidization (F 1) represent stresses that are nearly equal to each other. It is an advantageous property of the latter technique that it is simpler and it is not dependent on a commercial product such as the shaking apparatus.

Abrasion tests were also carried out with the fractions of the granulate heap. The fractions were kept in a fluidized state by an air stream at a threefold layer expansion for 10 minutes, sieved on a sieve corresponding to the lower dimension limit, and the residual material was measured. The value of the abrasive strength was defined in the following manner:

$$K_s = \frac{G_m}{G} \cdot 100 \qquad (12)$$

where

K_s is the abrasion strength (abrasion resistance) (per cent),

G is the mass of the weighed-in granulate fraction (gram),

G_m is the mass of grains remaining on the sieve corresponding to the lower dimension limit of the granulate fraction (gram).

The following values were obtained for the abrasion strength of the granulate fractions:

d = 0.25 - 0.40 millimetre K_s = 75 per cent
d = 0.40 - 0.63 millimetre K_s = 64 per cent
d = 0.63 - 1.00 millimetre K_s = 62 per cent
d = 1.00 - 2.00 millimetres K_s = 61 per cent

It can be concluded from these data that the abrasion strength of granulates larger than medium is approximately the same, and slightly decreases with increasing size; furthermore, the change in the amount of residual material on the sieve is greater even in the case of medium size than the decrease in average diameter when the whole granulate is subjected to abrasion. The strength of a large number of granulate-fractions was tested and this same tendency was observed in every case. Accordingly, it was concluded that for the evaluation it is sufficient to determine the abrasion strength of the granulate fraction of medium size, i.e. 0.4-0.63 millimetre.

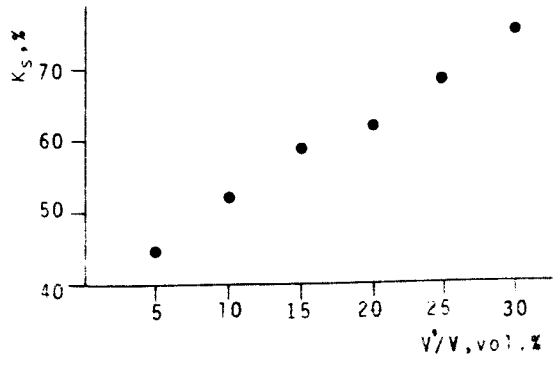

Fig.5

The abrasion strengths of the 0.4-0.63 mm fractions if six granulate heaps are shown, as an example, in Fig.5. The granulates were prepared by fluidization under identical circum-

stances and with the use of different relative amounts of liquid containing the same amount of binding agent. It is apparent from the Figure that the measuring method can be applied for studying the dependence of the abrasion strength on various parameters. The laboratory-type fluidization apparatus (cf. Fig. 3) needed and the procedure of measurement are simple and the results obtained are comparable, because the degree of mechanical stress is independent of the make of the apparatus, as is the case with various types of shaking apparatuses.

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РЕЗЮМЕ

Знание физических свойств гранулированных зерен важно с точки зрения как использования, так и научной исследовательской работы. Автором изложены методы измерения встречающиеся в литературе, пригодные к определению важнейших физических показателей гранулированных зерен (распределение размеров зерен, показатели плотности, доля объема пор, свойства истечения, прочность), а также дан отчет о результатах исследовательской работы, выполненной в связи с испытанием применимости методов измерения, и разработкой новых методов измерения. Автором введены понятия "плотность разрыхленных множеств зерен" и "коэффициент истечения", и указаны методы измерения применимые для их определения. Дан отчет о разработке простого метода измерения доли объема пор, основанного на одинаковые свойства заполнения пространства зерен подобной формы. Для определения прочности против износа (износостойкости) гранулированных зерен описан автором новый метод измерения, основанный на измерение действия механической нагрузки в псевдоожиженном слое.