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STUDIES ON GRANULATION IN FLUIDIZED BED IV.

EFFECTS OF THE CHARACTERISTICS OF THE FLUIDIZED BED THE ATOMIZATION AND THE AIR DISTRIBUTOR UPON THE PHYSICAL PROPERTIES OF THE GRANULATES

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It is important to know the influence of the characteristics of the granulator and the procedure upon the physical properties of the granulates produced, both from the point of view of the granulator design and the determination of the optimum operating parameters. However, few papers dealing with the above-mentioned questions are to be found in literature. The effect of the following factors: ratio minimum bed height to diameter of bed, degree of expansion of the fluidized bed, degree of dispersion of the granulating liquid, distance of the atomizer as measured from the air distributor and the type of the distributor upon the physical properties of the granulates produced was studied in a laboratory-size fluidized bed granulator of batch operation. In the evaluation of the experiments, the results are compared with the conclusions published in literature and the nearly optimum values for the above-mentioned variables are given.

From among the parameters having an influence upon the physical properties of granulates produced in a fluidized bed, the effect brought about by changes in the relative amount of the granulating liquid, the rate of addition of the latter, its concentration, and the total amount of binder fed in were investigated in the previous papers of this series [1, 2]. Approximative formulae were presented on the basis of those experiments for the calculation of the mean diameter of granules and the feed rate of the granulating liquid. The present paper deals with such characteristic parameters of the procedure and the apparatus that are of importance mainly in connection with the design and operation of fluidized bed granulators. These parameters are the following:

- ratio of the minimum bed height to the diameter of the bed;
- degree of expansion of the fluidized bed;
- degree of dispersion of the granulating liquid;
- distance of the atomizer from air distributor plate;
- performance of the air distributor plate.

As an introduction, conclusions of other authors in connection with the effect of the above-mentioned variables so far published in literature are summarized. The experimental results and conclusions drawn from them are then described.

In the design of fluidized bed granulators when deciding the size of the material container, or in the case of an existing apparatus, when determining the charge weight, the question of the preferable ratio minimum bed height to bed diameter arises. Simple fluid mechanical considerations also lead to the conclusion that this geometric simplex may have an optimum value or optimum value range. It is known from literature on fluidization [3, 4] that an increased danger of channel formation, (slugging) is encountered both with too shallow and with too high beds. These irregularities have an adverse effect on the mixing, heat and mass-transfer processes and also on the granulation. In addition, the ratio bed height to bed diameter may influence the probability of collision between the atomized liquid droplets and the particles, and also the rate of agglomeration [5]. In spite of the probably considerable influence of this variable on the physical properties of the granules produced, and of its economic importance (maximum utilization of the capacity of the apparatus), no paper so far published deals with this question in the manner that it deserves.

The degree of bed expansion is a very important characteristic of fluidization and its effect must be taken into consideration in all processes carried out by fluidization. In the case of fluidized bed granulation, an appropriate choice of the degree of bed expansion is of decisive importance regarding the final size distribution particle of the product [5, 6, 7, 8]. An increase in bed expansion tends to decrease the mean granule size [5, 8] because the more vigorous motion results increased abrasion. Another factor which makes higher bed expansion undesirable is an increase in elutriation. On the other hand, too small bed expansion brings about decreased mixing, heat and mass transfer, leading to the formation of "lumps" beyond the prescribed size limit. Accordingly, the optimum value of bed expansion is always a compromise between processes which are adverse as regards granulation and which tend to act against each other.

It is very difficult to determine accurately the degree of the dispersion of the granulating liquid, i.e. the size, and size distribution of the atomized droplets. Consequently, the effect of this parameter was studied only in an indirect manner. For example, in the case of two-fluid atomizers, which are most frequently applied in the practice of fluidized bed granulators, the effect of the pressure and flow rate of the atomizing air upon the physical properties of the produced granules were studied [8, 9]. The opinion of the different authors on this question is divergent. According to the paper of SCOTT and his co-workers [5], containing mainly theoretical considerations, it is to be expected that the mean drop size of the spray influences the process of granulation. The authors ascribe this to two facts: first, increasing the degree of dispersion also increases the specific surface area of the droplets and together with it the rate of evaporation of the latter. Secondly, the size, size distribution and number of atomized droplets influence the probability of collision between the droplets and solid particles, and thus also the rate of agglomeration. However, no experiments were carried out to study the influence of the degree of atomization. According to the work of MÖBUS [8], changing the pressure of the atomizing air between certain limits does not appreaciably influence the physical properties of the granules. Unfortunately, experimental results were not published and accordingly it is impossible to determine even the pressure range studied. Detailed experimental data have been published by DAVIES and GLOOR [9]. In contrast to the above-mentioned author, they found that upon increasing the atomizing air pressure from 0,5 to 2.0 kg/cm², the mean granule size was decreased from 438 to 292 μ.

The vertical distance of the spray nozzle from the air distributor is restricted between certain limits [5, 7]. The lower limit that can be taken into consideration is defined by the condition that the atomized liquid must not wet the distributor plate because this would lead to the formation of large lumps, clogging of the distributor and stopping of particle motion. The upper limit of the location of the atomizer is determined by the spray cone angle and the bed height, on the basis of the condition that the atomized granulating liquid should not wet the wall of the granulator. Opinions differ as to whether or not the variation of the height of the nozzle between the above-mentioned limits influences the physical properties of the granules. According to MÖBUS [8], changing the position on the nozzle does not appreciably influence the physical properties of the granules. On the other hand, various authors have observed that increasing the distance between the atomizer and the distributor brings about a decrease in the mean granule diameter [6, 9]. The change can be termed as considerable. For example, experiments carried out by RANKELL and his co-workers showed that the mean granule size was decreased from 500μ to half this value when the atomizer in a granulator, 0.3 m in diameter, was lifted from a height of 0.75 to 1.5 m [6]. The phenomenon was mainly explained by the following consideration: in the case of a highly positioned atomizer, the spray drying that is undesired from the point of view of granulation, become prevalent.

In the case of fluidized bed granulators, mainly perforated plates, sieves and porous plates are applied as distributors. A number of points simultaneously have to be taken into consideration when choosing the distributor. Economic considerations require a distributor plate that is simple, inexpensive, easy to procure, and causes a low pressure drop. Fluid mechanical considerations would require gas distributor plates made of sintered glass or metal, fireclay, or other similar porous materials, in order to obtain optimum fluidization motion and to avoid irregularities in the fluidization process [3, 4]. This question can be solved satisfactorily only if the influence of the quality of the distributor on the physical properties of the granulates produced is known. As far as the authors know, no other researchers have so far dealt with such studies.

Having reviewed the literature connected with the subject it can be stated that the parameters in question include some whose effect has yet not been studied in any way; in the case of others, mainly theoretical conclusions were drawn and the latter were checked by a few experiments only. In some cases the conclusions of various authors were contradictory to each other. To summarize it can be stated that the data in literature are insufficient to determine the approximately optimum values of the different parameters.

EXPERIMENTAL APPARATUS AND METHODS

A detailed description of the laboratory-size fluidized bed granulator used for the experiments and the experimental techniques will be dispensed with, because they are similar to those described in the second paper of this series [1]. The materials used in the experiments were a quartz sand fraction of $(0.1-0.2)\times 10^{-3}$m particle size and a granulating liquid which was an aqueous gelatine solution of $c' = 60$ kg/m³ concentration. The relative amount of the granulating liquid as referred to the total particle volume of the starting material to be granulated ($V'/V = 20$ % by volume), the feed rate of the granulating liquid ($w' = 5.9\times 10^{-5}$ kg/sec) and the input temperature ($T_b'' = 70$ °C) were kept constant.

EXPERIMENTAL RESULTS

The results of the research work carried out in order to study the influence of the technical and apparative parameters listed at the beginning of the present paper will now be described. The influence of changes in the individual parameters upon the mean particle size of the granulated material obtained, the mean porosity of the granules and the relative amount of the "product fraction" are illustrated in the Figures. Considering the conclusions on the reproducibility of the experimental results, published in the second paper of the series [1], three parallel experiments were carried out for each experimental point. The points drawn into the Figures represent the mean value of three such parallel results. It should be noted in connection with the Figures that the only reason to connect the experimental points with straight lines was to illustrate the trends of the changes.

a) Influence of Changes in the Minimum Initial Bed Height and Bed Expansion on the Physical Properties of the Granules

In the case of a given diameter of the granulator, the minimum bed height of the material to be granulated can be altered by changing the amount of the material fed in. In the first experimental series, the initial particle volume was increased from 200×10^{-6} to 600×10^{-6} m^3, in steps of 100×10^{-6} m^3. Simultaneously, the minimum initial bed height showed an approximately threefold increase. Of course, in proportion with increasing the amount of the material, the amount of granulating liquid was also increased, so as to have a constant relative amount of granulating liquid as referred to the particle volume of the material to be granulated.

Fig. 1 shows the changes in mean granule diameter and mean porosity of granulated material plotted against the minimum initial bed height. With increasing minimum initial bed height, the mean granule size shows at first an abrupt decrease and later on it converges to a limiting value. In addition, it is apparent that the change in mean granule size is strikingly large as the minimum starting bed height is increased from 6.4×10^{-2} to 8.7×10^{-2} m. The mean porosity of the granules closely follows any changes in the mean granule diameter. The relative amount of a granule fraction and any changes in this amount depending on the parameter under test are index numbers of considerable importance from the point of view of production. Fig. 2 shows the changes in the relative amount of the granules of the dimension $(0.2 \text{ to } 2.0) \times 10^{-3}$ m plotted against the minimum starting bed height. The weight ratio of the granule fraction conforming to the limits $(0.2 \text{ to } 2.0) \times 10^{-3}$ m is the highest (0.81 %) at a minimum starting bed height of 6.4×10^{-2} m. Further increasing the initial bed height is very disadvantageous from the point of view of this parameter.

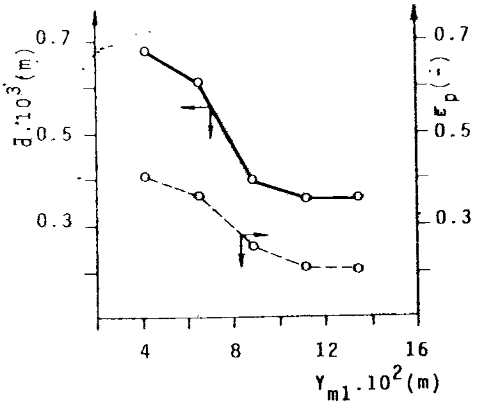


Fig. 1. $c' = 60$ kg/m^3
$V'/V = 20$ vol.%
$w' = 5.9 \times 10^{-5}$ kg/sec

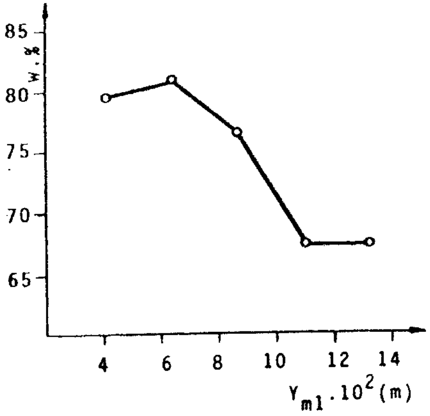


Fig. 2. $d = (0.2 - 2.0) \times 10^{-3}$ m

The degree of expansion of the fluidized bed can be characterized in the simplest way by the ratio height of the fluidized bed to the minimum bed height. During the granulation experiments, this ratio was approximately maintained at the predetermined value. With progressive agglomeration, the minimum bed height also changes (it generally increases). Accordingly, from time to time the air supply was stopped for a short period, the minimum bed height was determined, and the air flow rate was adjusted so as to obtain the same relative bed expansion.

In the experiments described so far, the ratio bed height to minimum bed height was the same ($Y/Y_m \approx 1.6$). In the study of the effect of bed expansion, the value Y/Y_m was increased from 1.3 to 2.5, in steps of 0.3, with the other parameters kept constant.

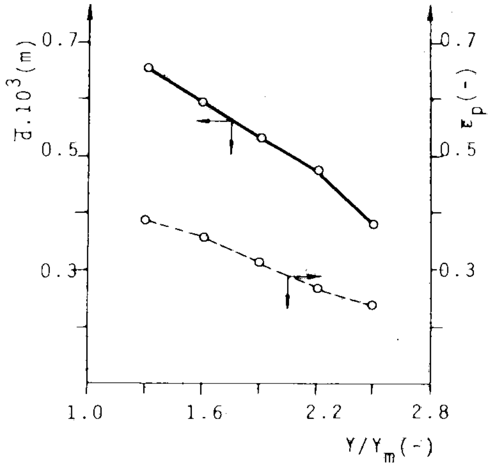


Fig. 3. $c' = 60\ kg/m^3$
$V'/V = 20$ vol.%
$w' = 5.9 \times 10^{-5}$ kg/sec

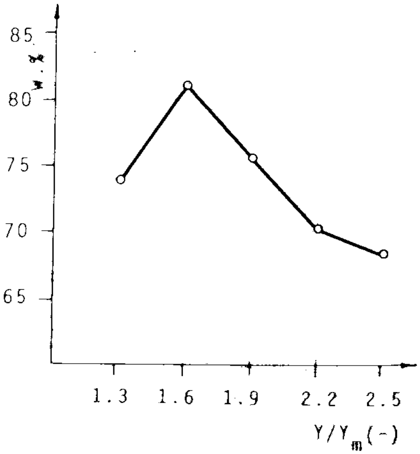


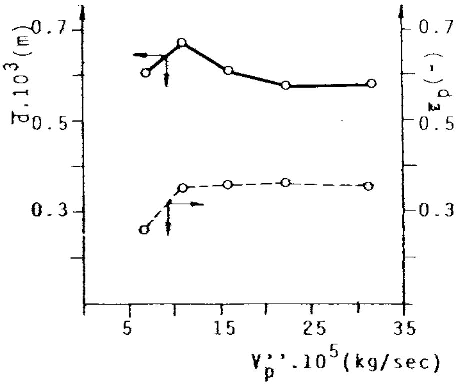
Fig. 4. $d = (0.2-2.0) \times 10^{-3}$ m

Fig. 3 shows the changes in the mean granule size and the mean porosity plotted against the degree of bed expansion. While increasing the ratio height of the fluidized bed to the minimum bed height from 1.3 to 2.5, the mean granule size showed a - nearly linear - decrease. Upon increasing the bed expansion, the mean porosity of the granulates also decrease nearly linearly in the range studied. It is apparent from Fig. 4 that the weight fraction of the granules of the $(0.2 \text{ to } 2.0) \times 10^{-3}$ m size range first increases and thereupon it gradually decreases with increasing bed expansion. The maximum value is at - or near to - a bed expansion of 1.6.

b) Influence of Liquid Dispersion and Location of the Atomizer on the Physical Properties of the Granules

In the case of the two-fluid atomizer used in these experiments, the degree of liquid dispersion can be influenced by adjusting the pressure of the atomizing air. With constant feed rate of the liquid to be atomized, the degree of liquid dispersion is increased by increasing the air pressure. In order to determine the connection between the two variables, it would have been necessary to measure the drop size distribution in the atomized stream, and the variations of the latter as a function of the air stream. However, such a detailed study of atomization was beyond the scope of the present experiments and it was considered sufficient, in order to be able to draw conclusions of a qualitative nature, to study the effect of the degree of liquid dispersion in an indirect manner, through the influence of the changes in the atomizing air stream.

In the next series of experiments, the only changed parameter was the air mass flow of the atomizer. The atomizing air flow in these experiments was increased from $(6.7 \text{ to } 30.5) \times 10^{-5}$kg/sec in four steps. It is apparent from Fig. 5 that the mean particle size of the granulated material is not influenced to an appreciable degree even if the atomizing air flow is changed considerably. The mean granule diameter changes in the $(0.57 \text{ to } 0.66) \times 10^{-3}$ m range: at first it increases and thereupon it slightly decreases. The mean porosity of granules shows an abrupt increase and afterwards its value remains constant. The relative amount of the granulated fraction corresponding to the size range $(0.2 \text{ to } 2.0)\times10^{-3}$ m changes according to a curve passing a flat maximum when plotted against increasing air atream - as shown in Fig. 6. However, it should be noted that when further increasing the atomizing air flow, at a value of 43.3×10^{-5} kg/sec, the experiment could no longer be evaluated. Accordingly, the changes in the physical properties of the granules brought about by the atomizing air stream are not as slow and gradual as could be judged on the basis of Figs. 5 and 6, but abrupt changes can be observed under a lower and over an upper limiting value. The explanation for this fact is that adequate dispersion of the liquid stops under a certain given air stream, whereas too fast an atomizing air stream virtually "shoots" the liquid into the layer and the material to be granulated clots unto the air distributor plate.



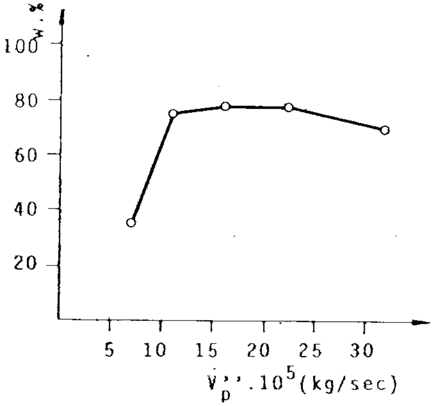


Fig. 5. $c' = 60$ kg/m³
$V'/V = 20$ vol.%
$w' = 5.9\times10^{-5}$ kg/sec

Fig. 6. $d = (0.2\text{-}2.0)\times10^{-3}$ m

The task of the next experimental series was to determine the effect of changes in the height of the atomizer on the physical properties of the granules produced. In these experiments, the vertical distance of the atomizer, as measured from the air distributor plate, was changed from 9.10^{-2} to 24.10^{-2} m in steps of 3.10^{-2} m.

At the same time, all other variables were maintained at a constant value. The mean particle size and mean porosity of the obtained granules plotted against the distance of the atomizer as measured from the air distributor is shown in Fig. 7, determined in these experiments. It is apparent from the Figure that the height of the location of the atomizer does not influence the mean granule diameter to a considerable degree within the studied range. The mean granule size value fluctuates between about 0.60×10^{-3} and 0.65×10^{-3} m. In this case, the mean porosity does not follow the changes in mean diameter, but it gradually increases with the increasing distance between the air distributor plate and the atom-

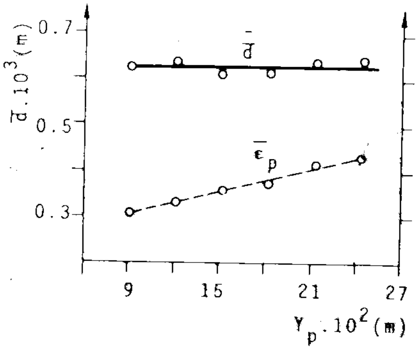


Fig. 7. $c' = 60$ kg/m^3
$V'/V = 20$ vol.%
$w' = 5.9 \times 10^{-5}$ kg/sec

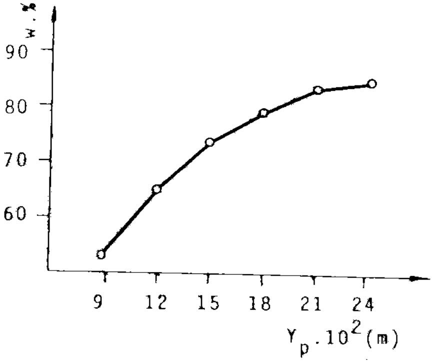


Fig. 8. $d = (0.2-2.0) \times 10^{-3}$ m

izer. Location of the atomizer has a most marked influence on the granule size distribution of the product. This is illustrated by Fig. 8 which shows changes in the relative amount of the granule fraction corresponding to the predetermined size range - $(0.2-2.0)\times10^{-3}$ m - plotted against the distance between the atomizer and the distributor. By lifting the atomizer from a height of 9.10^{-2} to 24.10^{-2} m, the amount of granules of product fraction was increased, according to the trend apparent from the Figure, from ∿ 53 % to ∿ 86 % by weight.

c) Influence of the Quality of the Air Distributor on the Physical Properties of the Granules

The influence of the quality of the distributor on the physical properties of the granules was studied with the application of four different distributors, under otherwise identical conditions. The distributor plates studied were the following: porous glass plates, sieves of 25×10^{-6} and 90×10^{-6} m openings, and a perforated plate of 12 mm perforations with a free surface ratio of 0.45; in order to prevent the escape of the granules, a sieve of 25×10^{-6} m openings was placed under the latter.

These experiments revealed the result that the mean granule size, mean porosity and particle size distribution of the granulated material obtained are practically independent on the quality of the air distributor.

EVALUATION OF THE EXPERIMENTAL RESULTS

According to the results of the present experiments, increasing the minimum starting bed height of the starting material decreases the mean granule size, and together with it, the mean porosity of the final product. An explanation for this phenomenon can be given - as has already been pointed out in the introduction - by two reasons. Increasing the starting bed height over a certain limit, this results in decreased uniformity of the fluidization, slugging and strong bubble formation being produced. The latter brings about an increase in the degree of regranulation. The second important effect of increasing the initial bed height in fluidized bed granulation is that - as far as the ratio of the wetted surface area of the bed to the total surface area is unchanged - increasing the bed height decreases the probability of the solid particles meeting a liquid droplet. In granulation tasks, it is generally required that as much of the product as possible should conform to the size limits defined by the purpose of application.

With this in mind, and on the basis of Fig. 2 and other experimental findings, it can be stated that in fluidized bed granulators it is preferable to choose an initial bed height that corresponds to 1/2 to 2/3 of the bed diameter (i.e. the inner diameter of the granulator). In the case of initial bed higher than that, the amount of particles left ungranulated will increase and the amount of the granules of product fraction will decrease.

In the case of a given starting material and a given binder, the degree of regranulation primarily depends on the intensity of the motion of the particles and on the degree of bed expansion. The decisive importance of this parameter regarding final granule composition was confirmed by the present experiments and is also in agreement with the findings of other authors [5, 6, 7, 8]. According to Fig. 3, the mean granule diameter and porosity show an abrupt decrease with increasing bed expansion, due to the increased abrasion of the granules. It can be concluded on the basis of Fig. 4 that the approximately optimum value is a relative bed expansion to about 1.6 times the initial value.

The experimental results illustrated in Figs. 5 and 6 justify the conclusion that the degree of liquid dispersion has, within certain limits, no appreciable influence upon the physical properties of the granules produced in the process. Increasing the atomizing air flow, primarily influences the particle size distribution however, as it is apparent from Fig. 6, in the middle part of the studied range, over a rather wide interval, even this influence is negligible. The amount of the "product fraction" is practically unchanged when the specific amount of atomizing air is increased to double its value, from 1.8 to 3.6 kg air/kg liquid.

The conclusions of the authors as to the influence of the degree of atomization agree with the opinion of MŎBUS [8] and disagree with the experimental results of DAVIES and GLOOR [9]. In addition to the different experimental conditions and techniques, this discrepancy can be explained by the fact that the drop size distribution of the atomized liquid, or any changes in it, were unknown in both cases and consequently the experimental results cannot be compared.

Some authors [6, 9] observed a decrease in mean granule size if the distance of the atomizer, as measured from the air distributor plate, was increased. In their opinion this is caused by the fact that the drops must travel a longer way to reach the bed if the position of the atomizer is higher. In this case, the smaller droplets may dry and lose their adhesive property. In the opinion of the authors of the present paper, whether the above-mentioned process - undesired with regard to granulation - does or does not occur, is determined not only, and not primarily by the position of the atomizer. Factors such as, e.g. the temperature of air leaving the bed, the concentration of the granulating liquid, and the fineness of the spray, etc., are responsible in this respect. The above process did not occur in the range studied in the present experiments and it can be stated on the basis of Fig. 7 that when changing the distance of the atomizer, as measured from the distributor, within the practically feasible range, the mean granule size remains unchanged. However, an interesting phenomenon can be observed on Fig. 7. This is the following: the higher position of the atomizer leads (even in the case of decreasing mean particle size) to the production of granules of higher porosity. This observation can be explained by the fact that increasing the distance of the nozzle from the air distributor plate, granule production can also occur in a bed of lower density, where the abrasive and compacting effects are less pronounced than in a fluidized bed of higher density. In the case of an unchanged cone angle of spray, the higher position of the atomizer increases the wetted surface area of the bed, i.e. the wetting is more uniform; and consequently the amount of granules of "product fraction" is increased (cf. Fig. 8). Of course, this can be true only up to a certain limit, since in the case of a too highly positioned nozzle, part of the granulating liquid wets the wall of the granulator instead of the bed as was pointed (out in the introduction), this is disadvantageous from several points of view.

On the basis of the results of these experiments and the experience on granulation acquired over several years, the following formula is proposed for the determination of the approximately optimum distance of the atomizer as measured from the air distributor plate:

$$Y_a = Y_{m1} + 0.8 \frac{D_b}{2 \ tg \frac{\alpha}{2}} \qquad (1)$$

Equation (1) expresses that the granulating liquid is to be atomized on top of the dense layer ($Y_d \approx Y_m$) in such a way - in order to prevent "carry up to the wall" - that the diameter of the circular wetted patch on top of the dense layer is 0.8 times the diameter of the apparatus.

The result obtained in connection with the influence of the type of distributor is of major importance. It can be concluded that it is unnecessary to apply distributors made of a porous plate, whose production on an industrial scale is difficult and expensive, and whose resistance against flow is high. The particle size distribution and other physical properties of the granulated material by the granulation process remain unchanged, if instead of a porous plate, a sieve of adequate mesh is used as a support. A perforated plate of large free cross sectional area is placed under the sieve to supply mechanical strength.

The results reported in the present paper were applied in the design of the pilot-plant and the industrial-scale fluidized bed granulator. The correctness of the design principles is confirmed by the fact that the physical properties of the granules produced by the equipment, correspond in every way to the predetermined standard.

SYMBOLS USED

c'	concentration of the granulating liquid (kg/m^3)
D_b	diameter of the apparatus (m)
$\bar{d}$	mean granule diameter (m)
d	particle size or sieve pore size (m)
T''_b	temperature of input air ($^{\circ}C$)
V	total particle volume of material to be granulated (m^3)
V'	volume of granulating liquid (m^3)
V''_p	atomizing air stream (kg/sec)
V'/V	relative amount of granulating liquid (vol.%)
w'	feed rate of granulating liquid (kg/sec)
Y	height of the fluidized bed (m)
Y_m	minimum bed height (m)
Y_{m1}	minimum starting bed height (m)
Y/Y_m	degree of bed expansion (dimensionless)
Y_d	height of the dense layer (m)
Y_a	distance of the spray nozzle, as measured from the air distributor (m)
α	angle of spray (degree)
$\bar{\varepsilon}_p$	average porosity of granules (dimensionless)

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РЕЗЮМЕ

В целях проектировании установок, гранулирующих посредством псевдоожиженного слоя, и определения оптимальных условий их работы важно знать какое действие оказывают технологические и аппаратурные характеристики на физические свойства образующихся гранул. В литературе встречается лишь незначительное число работ, подробно занимающихся этим вопросом. Авторы данной статьи на основании экспериментов, проведенных в условиях лабораторного реактора периодического действия с псевдоожиженным слоем, определили отношение минимальной высоты слоя/к диаметру слоя, зависимость размера слоя от скорости газа, а также то, в какой степени влияет на физические свойства гранул вид воздухораспределительной пластины и расстояние распылительной головки от этой пластины. Кроме того, авторы сравнили свои экспериментальные результаты с соответствующими литературными данными, и далее представили оптимальные значения вышеупомянутых переменных.