

Tadeusz GLUBA, Andrzej OBRANIAK, Estera GAWOT-MŁYNARCZYK*

THE EFFECT OF GRANULATION CONDITIONS ON BULK DENSITY OF A PRODUCT

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Changes in bulk density of a bed during wet granulation of silica flour in a batch drum granulator were studied. Three size fractions of the flour were used in the studies. They differed in the particle size composition and mean particle dimension. Variable parameters were the wetting conditions (droplet diameter, degree of bed saturation) and particle size composition of the raw material. The bed of loose material was wetted while tumbling, at a constant volumetric flow rate, using a system of two pneumatic spray nozzles. In each trial at determined time intervals feed samples were taken from the drum to specify the particle composition and bulk density of granulated material at a given stage of the process. The effect of droplet diameter, mean size of raw material particles and the saturation of granulated bed on changes in the bulk density of a product was estimated. A correlation equation describing the effect of tested parameters on bulk density changes was proposed.

Key words: drum granulation, bulk density

INTRODUCTION

One of significant parameters that describe properties of granular materials is their bulk density. For granulated materials this parameter is of special importance. By selecting proper conditions of granulation process a product of possibly high density can be obtained, which has an influence on a better utilisation of storage area and means of transport. Bulk density of a granulated product depends both on raw material properties (density, particle size composition), concentration of particles in the formed granules (granule porosity), and on the obtained particle size distribution on which the volume of intraparticle space depends [Gluba and Grabowski (2001), Podczeczek and Lee-Amies (1996)]. In the process of granulation bulk density of the processed bed undergoes systematic changes that are related both to the mechanisms of formation

* Technical University of Lodz, Faculty of Process and Environmental Engineering
Stefanowskiego 12/16, 90-924 Lodz, Poland, gluba@wiposp.lodz.pl

and growth of agglomerates, their concentration, and also the mechanisms of destruction. Problems related to the effect of granulation conditions on bulk density of a product are not frequently discussed in literature. Obraniak (2002) presented changes in bulk density of granulated material produced from foundry bentonite in reference to process and equipment parameters and wetting time. He obtained a linear relation of bulk density changes with the time of granulation. Zuurman et al. (1995) conducted studies on the effect of a binding agent on bulk density and compactibility of granules of two types of lactose, using two different wet granulation techniques. They found that efficiency of the binder increased with a decrease of bulk density of the granulated bed. Yu et al. (1995) studied the effect of moisture content on coal agglomeration and bulk density. They observed among the others that the density of agglomerates increased with an increase of moisture content to a certain maximum, and next it started falling down, while bulk density of the bed decreased with an increase of moisture content to some minimum and then it started growing.

So far, the bulk density of a granulated bed has been investigated for selected groups of materials, which made it impossible to propose general relations. Additionally, there are no studies in which the effect of other parameters (beside moisture content) that characterise bed wetting is taken into account.

AIM OF STUDIES

The aim of studies was to assess the effect of bed wetting conditions and particle size of raw material on changes in the bulk density of feed during wet drum granulation.

EXPERIMENTAL SET-UP AND METHODOLOGY

Granulation was carried out batch-wise in a horizontal drum of diameter $D=0.5$ m and length $L=0.4$ m. The drum was driven by an electric motor through a toothed gear and belt transmission. In the entire series of investigations a constant rotational speed of the granulator $n=20$ rpm was applied and a constant volumetric degree of drum filling with the raw material $k=0.1$, determined in reference to the bulk density of loosely packed material was used. A tested material consisted of three size fractions of silica flour from Strzeblowska Mine of Mineral Raw Materials at Sobótka. Particular fractions denoted by the symbols MK 0,056, MK 0,075 and MK 0,10 differed in the maximum particle size and range of particle size composition. For each raw material the basic physical properties were determined. The particle size distribution was estimated by means of a laser particle analyser ANALYSETTE 22, and on this basis the mean particle size d_z was determined. Raw material density ρ_s and bulk densities in the material loosely packed ρ_{bl} and concentrated to the minimum volume ρ_{bc} were also determined. Fine-grained material placed in the drum was wetted while tumbling by means of two pneumatic spray nozzles introduced axially into the drum. Constant

flow rate of the wetting liquid (distilled water) through the nozzles, equal to $Q_w=12 \cdot 10^{-3} \text{ m}^3/\text{h}$ was applied. Changes in the wetting liquid dispersion (droplet size) were obtained by changing the rate of air flow through the nozzles in the range $Q_p=1$ to $3 \text{ m}^3/\text{h}$. Droplet size distribution in the dispersed stream at specified parameters of nozzle operation, characterised by the dispersion degree $q=Q_w/Q_p$, was measured by a DANTEC laser analyser. On the basis of these distributions, the mean droplet diameters d_k obtained at given parameters of nozzle operation were calculated. The operating parameters of the nozzle used in the investigations are given in Table 1. Each time, a determined volume of the binding liquid was supplied to the bed. The volume was determined for a specified value of feed saturation degree S , taken as a ratio of the volume of the added liquid to the volume of intraparticle space in the loosely packed material in the drum. Granulation was studied at three degrees of saturation $S = 0.32, 0.34$ and 0.36 . After wetting the process of granulation was continued until the moment when water pressed from the granules onto their surface caused intensive sticking to the drum walls hampering in this way a further process. In determined moments samples were taken from the drum and on their basis properties of the formed granulated product were specified. The first sample was taken immediately after the wetting had been finished ($t_g=0$), and the last one after completing the process. The particle size composition was determined on the basis of screen analysis and bulk density was calculated on the basis of the mass and volume of a sample placed in a measuring cylinder (after subtracting the mass of water contained in it).

Table 1. Operating parameters of spray nozzles

Q_w	Q_p	q	d_k
$[\text{m}^3/\text{h}]$	$[\text{m}^3/\text{h}]$	$[-]$	$[\mu\text{m}]$
0.012	1.0	0.0120	235.70
0.012	1.5	0.0080	211.90
0.012	2.0	0.0060	194.11
0.012	2.5	0.0048	154.16
0.012	3.0	0.0040	143.90

RESULTS AND DISCUSSION

On the basis of analysis of samples taken immediately after finishing the wetting it was found that the bed contained both non-granulated raw material and a specified percent of nuclei and still weak granules. Mutual quantitative proportions between the finest fraction (of dimensions $< 1 \text{ mm}$), containing non-granulated material and nuclei of granules and bigger agglomerates appeared to be dependent on the bed wetting conditions and also on particle size of the raw material. Percentage of the smallest size fraction in the bed was in the range from 40 to 60%. Structure of the bed formed after

wetting had an immediate influence on its bulk density. The effect of particle size of the raw material and wetting parameters on the bulk density of the bed (after wetting) was described by the power function of several variables. As a result of regression, equation (1) with the correlation coefficient $R = 0.93$ was obtained:

$$\rho_{bn} = A \cdot d_k^{0,32} \cdot d_z^{0,37} \cdot S^{-1,8} \quad (1)$$

where A is constant.

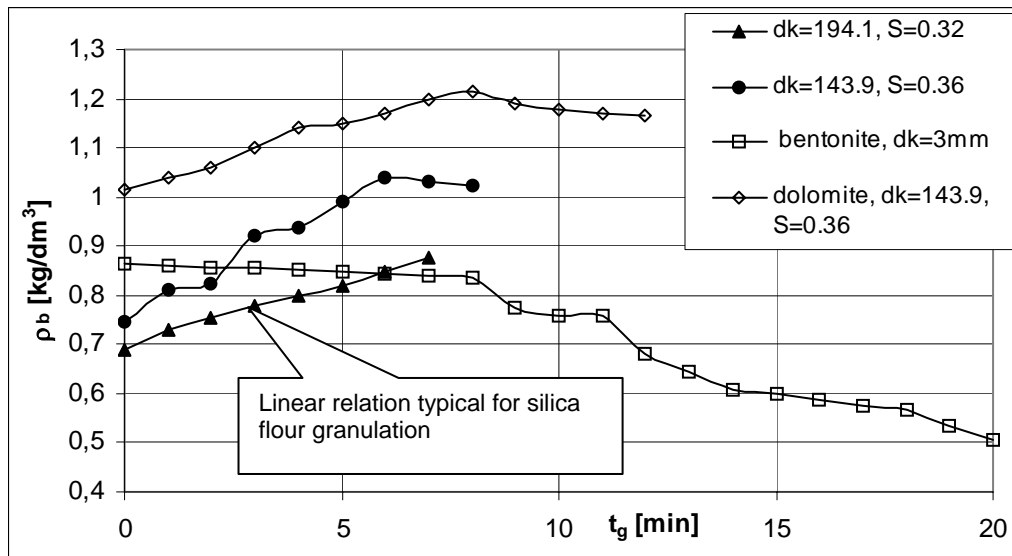


Fig. 1. Examples of changes in the bulk density of the bed during granulation

At the further stage of the process (granulation) systematic changes in the granulated bed properties took place leading to changes in bulk density of the bed. An example of changes in bulk density of the granulated bed of silica flour compared to the results obtained for other materials (dolomite and foundry bentonite) [Gluba (2003), Obraniak (2002)] is shown in Fig. 1. For most trials with silica flour a linear increase of bulk density with granulation time was obtained. In certain process conditions when a longer time of granulation was required, changes of bulk density in time departed from linearity (e.g. for $d_k=0.1439$). Analogous relations were obtained during dolomite granulation in similar wetting conditions. The required granulation time for this material was usually longer than for silica flour, which had an effect on the character of changes in bulk density (the presence of an extremum). A different character had the changes observed during bentonite agglomeration. In this case the process mechanism was different; in the whole granulation period wetting liquid in the

form of droplets with uniform size (about 3 mm) was supplied to the bed at low wetting intensity.

The differences observed in particular cases are a consequence of the dominance of various mechanisms of formation and growth of agglomerates at different stages of the process. To explain this problem some theoretical analysis should be made.

Bulk density of the feed during granulation can be determined on the basis of its mass m and volume V using the formula:

$$\rho_b = \frac{m}{V} \quad (2)$$

The mass of processed feed m is a sum of solid mass m_s and the mass of supplied wetting liquid m_w , while the volume of the bed consists of the volume of material particles V_s , binding liquid volume V_w and air volume V_p , hence:

$$\rho_b = \frac{m_s + m_w}{V_s + V_w + V_p} \quad (3)$$

The mass of raw material particles m_s is constant during the process, but the proportions between the mass of particles that form agglomerates and the mass of still non-granulated powder are changing continuously. A total volume of all particles in the bed V_s has also a constant value. The volume of air contained in the bed prior to wetting can be determined on the basis of the bulk density of a given raw material ρ_{bs} and specific density ρ_s . Changes in this volume during the process are very complex and difficult to describe because of a simultaneous interaction of many mechanisms that determine the formation, growth and also destruction of agglomerates.

From the practical point of view important is the bulk density of a dried product. In this case, when air of the same volume replaced the evaporated liquid, bulk density can be defined by the equation:

$$\rho_b = \frac{m_s}{V_s + V_w + V_p} = \frac{m_s}{\frac{m_s}{\rho_s} + \frac{m_w}{\rho_w} + V_p} \quad (4)$$

Substituting the relation for bed moisture content into equation (4)

$$w = \frac{m_w}{m_s} \quad (5)$$

form (6) is obtained:

$$\rho_b = \frac{1}{\frac{1}{\rho_s} + \frac{w}{\rho_w} + \frac{V_p}{m_s}} \quad (6)$$

Due to constant value of the final bed moisture content w (in a given trial) and raw material mass m_s and taking into account that specific powder density ρ_s and water density ρ_w do not change during the process, the above relation shows that changes in bulk density of the granulated bed ρ_b at the stage of granulation depend only on changes in the volume of air contained in the bed V_p .

To determine monotonicity of the above relation, the differential should be calculated:

$$\frac{d\rho_b}{dt} = \frac{-1}{\left(\frac{1}{\rho_s} + \frac{w}{\rho_w} + \frac{V_p}{m_s}\right)^2} \cdot \frac{1}{m_s} \cdot \frac{dV_p}{dt} \quad (7)$$

In order to set up the sign of this relation the differential dV_p/dt should be analysed.

The air in the granulated bed can be contained in the formed granules (V_1), between these granules (V_2) and between particles of still non-granulated raw material (V_3).

Hence:

$$\frac{dV_p}{dt} = \frac{dV_1}{dt} + \frac{dV_2}{dt} + \frac{dV_3}{dt} \quad (8)$$

Owing to the fact that the percentage of non-granulated particles decreases with the process, the value of V_3 decreases which brings about a negative value of derivative of this component after the granulation. Along with the granulation time the formed agglomerates are concentrated which results in a systematic decrease of pore size inside the granules leading to negative dV_1/dt . Changes in the volume V_2 are a result of changes in granule compactibility in the bed which depends on its homogeneity. Results of investigations on drum granulation obtained in this study as well as those presented by other authors [Gluba and Heim (2000)] show that with the granulation time grows monodispersity of the processed bed (Fig. 2) which causes that spaces between the formed granules increase. This is confirmed by the presented relation of changes in bed homogeneity coefficient s/d with the granulation time (where s – standard deviation, and d – mean particle size). It can be observed that despite decreasing values of the coefficient s/d (the bed homogeneity increase) bulk density determined for both dry and wet product increases.

Summing up the above considerations, the character of the effect of particular volume components can be presented in the form of inequality:

$$\frac{dV_2}{dt} > 0, \quad \frac{dV_1}{dt} < 0, \quad \frac{dV_3}{dt} < 0 \quad (9)$$

Taking the above into account, it can be stated that during the process of wet tumbling granulation changes in the bulk density may be both increasing and decreasing – depending which of the mentioned derivatives has a decisive influence in a given stage of granulation. So, it can be concluded that the character of changes depends both on the loose material properties and process parameters. To explain the impact of particular parameters applied in the investigations the obtained results should be analysed thoroughly.

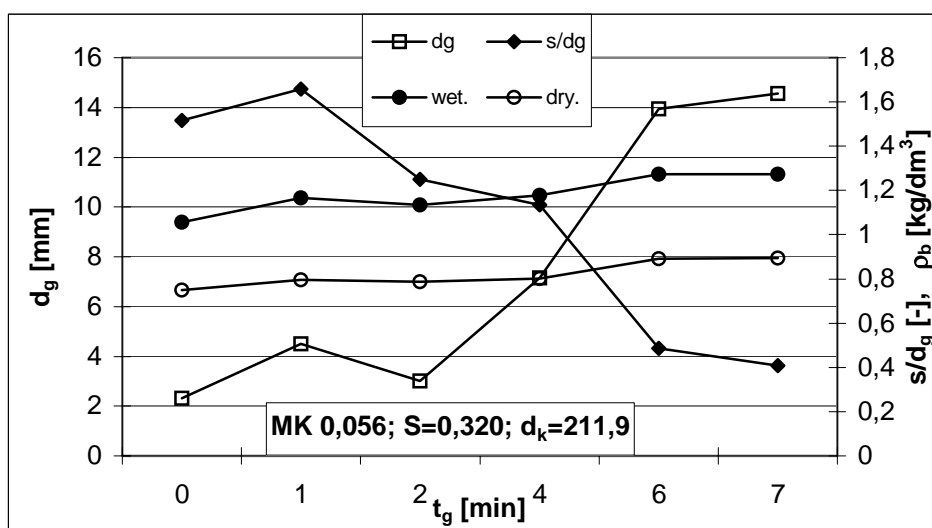


Fig. 2. Comparison of changes in bulk density of wet and dry granular material, variability coefficient and mean granule diameter during granulation

When considering changes of the bulk density as a function of granulation time (Fig. 3), for materials of different particle size composition, at constant saturation and the same droplet size it was found that for the materials with bigger particles, the bulk density is higher. This character of the obtained relations is related both to the compactibility of the particles in the formed granules, to the packing of the granules in the bed, and to the mean distance between the particles of non-granulated raw material. Fine-grained materials used in the study were characterised by a growing range of particle size composition with an increase of the mean particle size (a simultaneous growth of d_z and s/d_z). For these materials, with an increase of the mean

particle size, it is possible to reach denser packing of particles in the granules, which is followed by a general increase of the bed bulk density. The study has shown that during the process of granulation there is a systematic unification of the particle size composition (a decrease of variability coefficient s/d_g) which in turn has an effect on the compactibility of granules in the bed. It was observed that with an increase of the mean size of raw material particle the bed in the same process conditions becomes less homogeneous (s/d_g increases). This means that the bed obtained from the material of bigger particles is characterised by a denser packing of granules, which influences its bulk density increase.

Results of the studies revealed also a significant effect of dispersion of the wetting liquid stream (droplet size) on the process of granulation and consequently on the bulk density of the processed bed. A comparison of changes in the bulk density of feed during the process of granulation for different mean size of droplets is shown in Fig. 4. It follows that the feed bulk density increases with an increase of wetting liquid droplets [Gluba (2002)]. Bigger droplets enable formation of stronger liquid bridges which as a result leads to bigger condensation of particles in the granules. Additionally, the bed wetted with bigger droplets is characterised by higher polydispersity. These two factors have an influence on the increase of bulk density of the bed with an increase of droplet size during wetting.

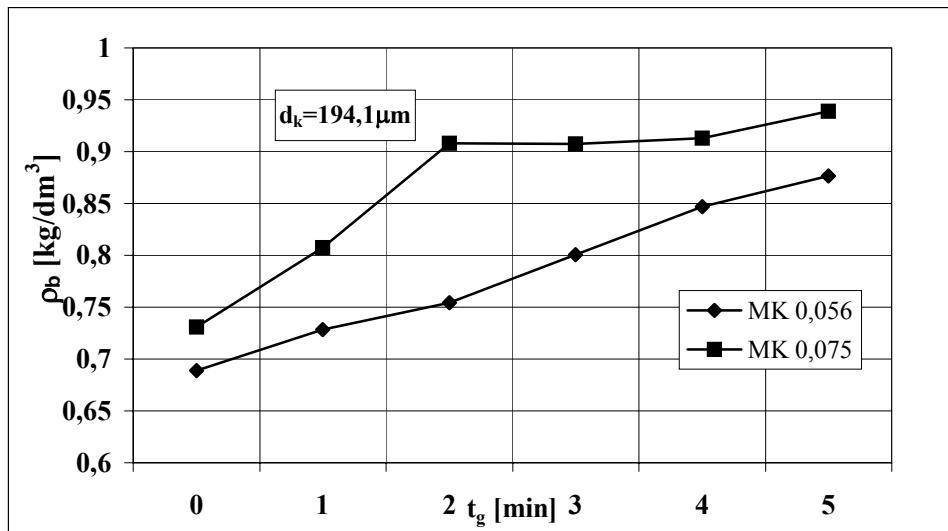


Fig. 3. Comparison of bulk density changes during granulation for the same droplet size and two types of raw material ($S = 0.320$)

On the stage of granulation, after wetting, continuous transformations induced by the mechanisms of formation, growth and destruction of agglomerates take place in the bed. A result of these transformations is an increase of the size of granules,

condensation of their inner structure and change in the particle size composition. These transformations being a determined function of granulation time lead also to specific changes in the bed bulk density. The change in bulk density of the bed in time is a combination of these transformations of the bed properties dependent on the properties of raw materials and process parameters.

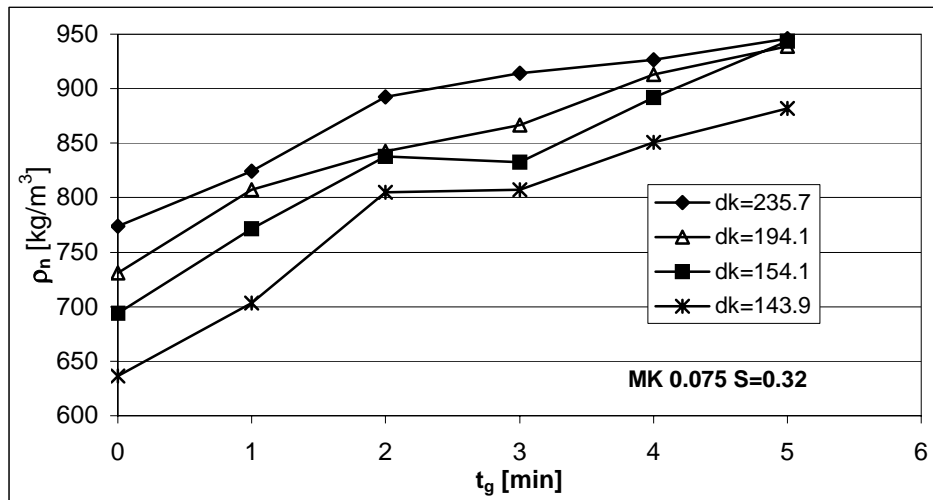


Fig.4. Comparison of bulk density changes during granulation for different droplet sizes

The effect of tested parameters on the increment of the bed bulk density at the stage of granulation is described by the power relation:

$$\rho_b - \rho_{bn} = B \cdot d_k^{0,3} \cdot d_z^{3,5} \cdot t \quad (10)$$

where B is constant.

The above relation was obtained at the correlation coefficient $R = 0.96$.

CONCLUSIONS

1. During wet drum granulation of silica flour, bulk density of the processed bed increases linearly in time;
2. The value of bulk density after wetting depends on the conditions of bed wetting (dimensions of the wetting liquid droplets and bed saturation) and on the size of raw material particles;
3. An increase of bulk density of the bed during granulation depends on the size of raw material particles and wetting liquid droplets;
4. The effect of tested parameters on the increase of bulk density of the bed during the process can be described by the power equation.

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Przeprowadzono badania zmian gęstości nasypowej podczas procesu granulacji mączki kwarcowej w granulatorach bębnowych. W badaniach parametrami zmiennymi były: parametry nawilżania tzn.: średnica kropli, współczynnik saturacji oraz skład ziarnowy surowca. Złoże materiału sypkiego nawilżano kropłowo w czasie jego ruchu przesypowego, przy stałym objętościowym natężeniu dopływu cieczy, za pomocą zestawu dwóch dysz pneumatycznych zapewniającego równomierne podawanie cieczy zwilżającej. Podczas każdej próby mierzono w stałych odstępach czasowych skład ziarnowy i gęstość nasypową granulatu na danym etapie procesu. Przeprowadzono ocenę wpływu średnicy kropeł, średniego wymiaru surowca oraz saturacji granulowanego złoża ziarnistego na zmianę gęstości nasypowej produktu. Zaproponowano równanie korelacyjne opisujące wpływ w/w parametrów na zmianę gęstości nasypowej.