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A MODEL OF GRANULE POROSITY CHANGES DURING DRUM GRANULATION

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A model equation to determine mean inner porosity of granules ε is proposed. The equation is based on the size analysis of bed particles (agglomeration kinetics) and the measurement of bulk density of feed. Bulk density of a granulated product depends on properties of the raw materials (density, particle size distribution), properties of the wetting liquid, particle concentration in the formed granules (porosity of the granules), as well as on the obtained particle size distribution on which interparticle space volume depends. In order to verify the proposed model, the process of drum granulation was tested for the following range of changing parameters: drum diameter D = 0.25 to 0.4 m, filling of the drum with granular material k = 5 to 20% of the inner volume, relative rotational speed of the drum $n_w = 0.15$ to 0.375 of critical velocity. The granular bed was wetted drop-wise during the drum rotations, at constant liquid supply rate Q = 1 cm³/s. During the process, at constant time intervals, samples were taken from the drum and on their basis values of bulk density of the feed as well as its particle size distribution, and inner porosity of the granules for a given size fraction were determined. The volume of liquid accumulated in the inner pores of the granules was specified basing on the mass balance before and after drying. Results of the research confirmed validity of the proposed model.

keywords: agglomeration, bulk density, drum granulation, porosity

1. INTRODUCTION

The process of tumbling granulation is usually described basing on the analysis of its kinetics (Kapur and Fuerstenau, 1966; Newitt and Conwey-Jones, 1958) The particle size distribution of the processed bed does not provide information on its

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many important properties, but on the other hand it determines the choice of some statistical parameters which characterise selectively the properties of tested granulated product. Heim et al. (2002) claimed that the parameter which well described granular bed properties was its bulk density ρ , a parameter easy to measure and frequently used in practice. This parameter, however, does not reflect either many significant features of agglomerates being formed. Additionally, it is not known if changes in bulk density of the granulated material are a result of changes in packing of individual granules in the bed, or changes in the porosity of granules as such. The porosity well characterises the structure of formed granules and could supplement the product description but unfortunately it is difficult to determine, especially online. For this reason, a new model of tumbling agglomeration was proposed to enable estimation of mean porosity of granules based on the measurement of bulk density of the granulated bed and its particle size analysis.

2. AIM OF THE WORK

The aim of this research was to develop a mathematical model of fine-grained bed granulation while wetting in a rotary drum. The model combines the agglomeration kinetics with changes in bulk density of the bed and porosity of granules in reference to air content in the processed bed.

3. EQUIPMENT AND INVESTIGATION RANGE

A schematic diagram of the experimental rig is shown in Fig. 1. Drum (1) was driven by motoreducer (3) through a belt transmission and a coupling. The rotational speed of the drum was changed smoothly using inverter (4). A granular bed placed in the drum was wetted drop-wise by means of sprayer (2), inserted axially to the drum.

The wetting liquid was supplied at constant flow rate $Q = 1 \text{ cm}^3/\text{s}$ from tank (6) placed on the level of 2.5 m from the drum axis. The liquid flow rate was settled by means of rotameter (7). The sprayer was mounted on a separate stand (5). For the whole time of the experiment a constant level of liquid in the tank was maintained which ensured constant pressure of liquid supplied. The wetting liquid was distilled water.

The process of granulation was carried out batch-wise in drum granulators of the same length L = 0.24 m and diameters ranging from D = 0.25 to 0.4 m. The filling of the drum with granular material k was changed from 5 to 20% of the inner volume, and relative rotational speed of the drum n_w from 0.15 to 0.375 of the critical velocity.

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Fig. 1. Schematic diagram of the experimental rig

4. MEASURING METHODS

In preliminary studies basic properties of the experimental material (foundry bentonite) were identified. Particle size distribution of the row material was determined using a FRITSCH laser particle size analyser and on this basis an average size was calculated. The size of bentonite particles was <0.16 mm, and their mean volumetric size was $d_m = 0.056$ mm. Density of the material measured by a pycnometer, was $\rho_s = 2150$ kg/m³, and mean bulk density determined as an arithmetic mean from the bulk density of material both loose and concentrated to a minimum volume in a shaker, was $\rho_{ns} = 865$ kg/m³.

To identify properties of the granulated product obtained in the process, representative samples were taken from the drum at constant time intervals (every 60 or 120 s). On this basis bulk density and particle size distribution of the feed were specified. After making appropriate measurements the sample was returned to further granulation. Based on the results of sieve analysis, mass fraction with particles $< d_o = 1$ mm was estimated. The fraction consisted mainly from not granulated particles of the raw material. For the entire process duration the instantaneous values of torque were measured every 1 s on the granulator shaft.

5. MODEL

Based on the analysis of a definition of bulk density Gluba et al. (2004) related changes in the bulk density of a granulated bed during wetting to moisture content of the feed w, density of raw material ρ_s and wetting liquid ρ_w , mass of raw material m_s and the volume of air contained in the bed V_p . The following relation (Eq.1) was obtained:

$$\rho = \frac{1 + \frac{m_w}{m_s}}{\frac{1}{\rho_s} + \frac{m_w}{\rho_w \cdot m_s} + \frac{V_p}{m_s}} = \frac{1 + w}{\frac{1}{\rho_s} + \frac{w}{\rho_w} + \frac{V_p}{m_s}},$$
(1)

where m_w is the mass of binding liquid, kg. The feed mass in the granulator m_s , specific density of powder ρ_s and wetting liquid density ρ_w do not changed in the process.

The above relation shows that changes in bulk density of the granulated bed ρ depend only on its moisture content w and the volume of air accumulated in the bed V_p . At continuous liquid supply, moisture content of the bed is directly proportional to time, and the only unknown value is air volume V_p . This volume is related to inner porosity of formed granules (V_{pg}), and to the volume of pores between the granules and unprocessed feed particles (V_{pmg}).

Taking this into Eq. 1, we obtain:

$$\rho = \frac{1+w}{\frac{1}{\rho_{s}} + \frac{w}{\rho_{w}} + \frac{V_{pg}}{m_{s}} + \frac{V_{pmg}}{m_{s}}}.$$
(2)

Further considerations were preceded by some simplifying assumptions. It was assumed that each droplet falling down on the bed formed a nucleus that accumulated wetting liquid and during the further process its dimension increased. This means that during wetting the whole water supplied to the bed was accumulated in the nuclei or granules being formed. Additionally, it was assumed that the considerations would refer to this stage only at which granulated product was formed, when not granulated material still remained between the granules, although with time its amount was decreasing. To describe the mechanism of changes in the volume of air accumulated inside the granulated bed and in the formed granules, the process of batch granulation was analysed at continuous wetting, from the initial point, i.e. dry powder material, until the moment when all raw material will become the form of granules.

The process discussed in the paper was initiated by sprinkling the bed which tumbled in a horizontal rotary drum. Liquid droplets of dimension about 3 mm fell down onto free surface of the tumbling bed inclined due to drum rotations, penetrated it and formed nuclei of future granules. At the beginning of the process, when only a few droplets have entered the bed, in the powder material single granules (nuclei) appeared, which were not in contact with one another. They formed single agglomerates suspended in a powder medium. When the process proceeded, the number of granules increased at the cost of unprocessed material, however for a fairly long time they constituted a few centres separated from each other by the powder material. At this stage of the process the granules were quite dry on the surface. This lasted until the moment when an increase of the number and size of the agglomerates led to a situation when individual granules got in contact, although spaces between them were still filled up by material that had not been granulated yet. A continuation of the agglomeration process resulted in a gradual granulation of the whole powder material, at a simultaneous increase of dimensions of the previously formed granules.

The initial air volume V_{pp} accumulated in the dry bed of feed, can be determined by subtracting from the total volume of the powder bed V_{cs} the volume of feed particles (bentonite) V_s , which formed this bed:

$$V_{pp} = V_{cs} - V_s = \frac{m_s}{\rho_{ns}} - \frac{m_s}{\rho_s} = m_s \left(\frac{1}{\rho_{ns}} - \frac{1}{\rho_s}\right).$$
 (3)

Due to the fact that with the formation of subsequent granules the fraction of not granulated material (bentonite powder) decreased, the above relation could be applied after taking into account the fraction of not granulated material U_m in any time interval (arbitrary bed moisture content) of the discussed granulation period. Then, we got the following relation:

$$V_{pmg} = U_m \cdot m_s \left(\frac{1}{\rho_{ns}} - \frac{1}{\rho_s}\right). \tag{4}$$

Substituting it to (2), the following form of bulk density relation is obtained:

$$\rho(w, U_m) = \frac{1+w}{\frac{1}{\rho_s} + \frac{w}{\rho_w} + \frac{V_{pg}}{m_s} + U_m \left(\frac{1}{\rho_{ns}} - \frac{1}{\rho_s}\right)}.$$
(5)

Fraction U_m is a function of bed moisture content. Upon transformation we get:

$$V_{pg} = m_s \cdot \left[\frac{1+w}{\rho(w, U_m)} - \frac{1}{\rho_s} - \frac{w}{\rho_w} - U_m \left(\frac{1}{\rho_{ns}} - \frac{1}{\rho_s} \right) \right].$$
(6)

If then a bed is formed from porous particles, e.g. granules composed of solid particles (Fig. 2), the total bed porosity depends both on the volume of intragranular spaces and pore volume in the granules themselves. Inner porosity of granules ε can be determined from the relation:

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$$\varepsilon = \frac{V_{pg}}{V_g} = \frac{V_{pg}}{V - V_{ns}},\tag{7}$$

where: V_g – total volume of granules, m³. Substituting Eq. (6) to (7) we have:

$$\varepsilon = \frac{m_s \cdot \left[\frac{1+w}{\rho} - \frac{1}{\rho_s} - \frac{w}{\rho_w} - U_m \left(\frac{1}{\rho_{ns}} - \frac{1}{\rho_s}\right)\right]}{V - V_{ns}}.$$
(8)



Fig. 2. Schematic diagram of a granular system formed by porous particles

The volume of not granulated raw material in the process moment V_{ns} is calculated from:

$$V_{ns} = U_m \cdot V_s = U_m \cdot \frac{m_s}{\rho_{ns}} \tag{9}$$

and

$$V = \frac{m_w + m_s}{\rho} \tag{10}$$

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hence, after transformations, we obtain the equation:

$$\varepsilon = 1 - \frac{\frac{(1 - U_m)}{\rho_s} + \frac{w}{\rho_w}}{\frac{1 + w}{\rho} - \left(\frac{U_m}{\rho_{ns}}\right)}.$$
(11)

6. RESULTS AND DISCUSSION

The results of our studies shown in Fig. 3 reveal that at the stage of granule formation, the mean porosity of agglomerates calculated from model relation given in Eq. 10 does not change with wetting time (bed moisture content).



Fig. 3. Changes in the mass fraction of not granulated bed U_m , bulk density ρ , mean granule porosity ε and reduced moment M^* as a function of moisture content

When analysing the mechanisms of granulated material formation at the discussed stage of the process and taking into account the type of wetting, it can be stated that the results obtained reflect with high probability the properties of produced agglomerates. It can be expected that the granules formed at the beginning of the process, which are first suspended in the powder material and then are enveloped in it, due to the lack of mutual interactions, do not condense and until the end of this stage their porosity is approximately constant. Measurements of bulk density of the granulated bed ρ during the process (with the growth of feed moisture content), shown for instance in Fig. 3, confirmed that changes of this parameter were decreasing linearly. Similar tendencies were observed by Heim et al. (2002a). Online measurements of torque changes carried out during the granulation process (Fig. 3) confirm conclusions presented by other authors (Heim et al., 2002b) that the torque is an indicator of process realisation. When analysing relations illustrated in Fig. 3, it was found that the upper limit of the feed moisture content for the range assumed in the discussed model corresponded to a maximum value of the reduced moment. In this range the mass fraction of not granulated raw material U_m decreased linearly with the increase of moisture content.

To generalise this conclusion, changes of U_m were analysed for all granulation trials performed (Fig. 4). The results obtained in the assumed range were approximated by the linear function:

$$U_m(w) = -a \cdot w + 1$$
, where a>0. (12)



Fig. 4. Changes in mass fraction of not granulated bed U_m as a function of moisture content for all experimental runs

This relation describes changes of U_m during drum granulation in a broad range of changes in drum diameters, rotational speed and degree of drum filling, but it refers only to the agglomeration of a selected material, i.e. bentonite. It is also limited by the range of moisture content (the first period). When the results are to be generalised to other powder materials, a universal form of linear Eq. 12 should be assumed.

The form of relation (Eq. 11) and a linear character of changes in the mass fraction U_m and bulk density of the bed ρ allow us to expect that in the discussed range of

bentonite granulation the effect of bed moisture content on mean inner porosity of the agglomerates ε is reduced. A consequence of this can be a constant value of mean porosity of agglomerates ε calculated from the model equation.

Completion of the presented model with relations characteristic for the second stage of granulation (the growth of agglomerates and their condensation) will enable a quick identification of changes in the properties of granules at any time of the process.

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Zaproponowano modelowe równanie do wyznaczania średniej wewnętrznej porowatości granulowanych materiałów. Równanie to oparto o analizę kinetyki agregacji oraz pomiary gęstości materiału poddawanego agregacji. Gęstość zgranulowanego produktu zależy od właściwości nadawy (gęstość, skład ziarnowy), właściwości cieczy zwilżającej, koncentracji ziarn w utworzonych granulach (porowatości), jak również od składu ziarnowego otrzymanych granul, od którego zależy objętość pustek. W celu zweryfikowania modelu, testowano proces granulacji bębnowej dla następujących parametrów: średnica bębna D = 0.25 do 0.4 m, wypełnienie bębna z granulowanym materiałem k = 5 do 20% objętości wewnętrznej, względna prędkość obrotowa bębna nw= 0.15 do 0.375 wartości krytycznej. Granulowane złoże było zwilżane kroplami cieczy w trakcji obrotu bębna przy stałej szybkości

wkraplania Q = 1 cm3/s. Podczas procesu, przy stałych interwałach czasowych, pobierano próbki z bębna i na podstawie gęstości materiału nadawy i jej składu ziarnowego wyznaczano wewnętrzną porowatość granuli dla danej klasy ziarnowej. Objętość cieczy akumulowanej w porach wewnętrznych granul były specyfikowane w oparciu o bilans masowy przed i po suszeniu. Wyniki badań potwierdziły ważność proponowanego modelu.

słowa kluczowe: aglomeracja, gęstość, granulacja bębnowa, porowatość