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NUCLEATION AND GRANULE FORMATION DURING DISC GRANULATION PROCESS

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Abstract. The study of nucleation and granule formation in a bed of fine-grained material during the phase of batch dumping in the process of periodical disc granulation was conducted. The study was carried out on laboratory disc (diameter - 0.5 m), with the use of constant disc filling with powder material, constant slope angle 50° between the disk axis and level and constant rotational speed of device. Foundry bentonite was used as the examined material and distilled water as a binding liquid. The binding liquid was added in the form of droplets having constant size. The size of droplets was changed in the range 2.4÷4.9 mm. The influence of number and size of binding liquid droplets delivered to bed, as well as of their delivery height on the size distribution of nuclei generated in the dumping phase was determined.

keywords: nucleation, disc granulation, size distribution, drop impact energy

1. Introduction

The flow of liquid through porous substances poses a serious problem, solution of which is necessary to carry out some operations taking place in three-phase systems such as granulation. Wet granulation is characterized by the participation of two disperse phases: grained powder and liquid phase introduced in the form of droplets and compact phase – the air. Their interaction, which depends on the degree of both phases dispersion, determines the progression of a given operation.

Wet granulation processes may be divided into three main stages: wetting and nucleation, consolidation and growth, and granule attrition and breakage (Ennis and Litster, 1997; Tardos et al., 1997; Iveson et al., 2001; Litster and Ennis, 2004). During the wetting and nucleation stage, the liquid binder is sprayed onto the tumbling fine-grained material (in rotary drums, disks) where it wets the powder and forms granule nuclei. The conditions of liquid distribution have a great effect on nucleation and final properties of the granule produced (Gluba, 2002; Hapgood et al., 2003; Gluba, 2003).

The phenomena of liquid drop impacts on powder beds plays an important role during nucleation in wet granulation processes.

When the drop size is larger than the particle size, wetting the powder with the liquid gives a distribution of seed granules or nuclei. When the drop size is small compared to the unit particle size, the liquid will coat the particles. The coating is produced by collision between the drop and the particle followed by spreading of the liquid over the particle surface.

If the liquid drop breaks on impact, then this will effectively increase the number of granule nuclei formed and decrease their size. If the liquid drop spreads out a large amount before it soaks into the powder, then this will increase the surface coverage term and may prematurely cause nuclei coalescence and caking at the powder surface.

Only a few studies concern the phenomena occurring during the collision of liquid droplets with the surface of the porous material powder (Agland and Iveson, 1999; Hapgood et al., 2002; Nguyen et al., 2009; Tan et al., 2009). However, most of them relate to other research systems and there is no simple transfer to the conditions of nucleation in wet granulation process.

2. Aim of the study

The aim of this study was to determine the effect of conditions for the supply of binding liquid droplets to bed in a disc granulator on the formation and growth of granule nuclei.

3. Experimental

Investigations of binding liquid droplet penetration in a moving bed of powder material located in a disc granulator were carried out on an experimental stand the scheme of which is shown in Fig. 1.

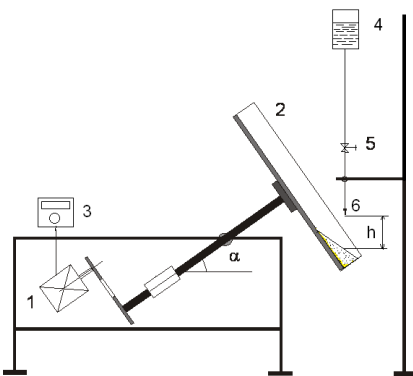


Fig. 1. Schematic diagram of the equipment.
1. engine with reducer, 2. disc, 3. inverter,
4. tank, 5. valve, 6. Needle. h - height of the needle
tip above the bed, α - angle of disk axis
inclination

Disc granulator (2) of diameter 0.5 m and height of the rim 0.1 m was mounted on a rotating shaft, inclined at an angle $\alpha = 50^\circ$. A shaft was driven by an electric motor with reducer (1) and inverter (3). During the tests a constant rotational speed of plate

was equal to 10 rpm and constant mass of fine-grained material equal to 0.3 kg. A wetting liquid was provided drop-wise from a dropping funnel (6) supplied from the tank (4) on a bed in the disc. The assumed value of the liquid flow intensity was determined by means of a valve (5). Binding liquid was supplied drop wise of various fixed size resulting from the size of applied funnel tips. The size of droplets generated for each tip was determined based on the weight and the number of drops supplied at a given time. The calculations assumed spherical shape of drops.

Three different heights of the binding liquid droplets supply resulting from the location of the funnel outlet tips at different heights h above the bed.

The parameters of the wetting liquid supply to the bed are summarized in Table 1.

Table 1. The wetting parameters used in the investigations

Hight of the needle tip above the bed, h [m]	Drop diameter d_d [mm]	Drops number n_d [-]	Liquid flow rate Q_w [cm ³ /s]
0.02 0.52 1.02	3.0, 3.3, 4.9	100, 200, 300	0.043
0.02 0.52 1.02	2.4, 3.3	100, 200, 300	0.025

A model experimental material was foundry bentonite. The basic properties of the raw material were: density $\rho = 2150 \text{ kg/m}^3$, mean bulk density $\rho_b = 865 \text{ kg/m}^3$, mean volumetric size $d_m = 0.013 \text{ mm}$. Distilled water was used as the wetting liquid.

Fine-grained material bed in the disc was given a specified number of liquid drops n_d and then the unit was stopped and obtained feed was separated into size fractions using a set of sieves. For each size fraction mass m_n and the number of granule nuclei n_n which formed in the course of dispensing drops of liquid to the bed were determined. At the same conditions of liquid supply a second attempt was performed each time during which after the supply of liquid to bed the process was continued over a fixed time $t_g = 10 \text{ min}$. The feed obtained after this time period was separated into size fractions and weight of each fraction m_{n10} and the number of granules in a given size class n_{n10} were determined.

4. Results

The results of this study indicate that the conditions for the supply of binding liquid to the powder bed significantly influence the penetration of the liquid in the bed and the associated process of creating granules nuclei. Depending on the size of dispensed droplets, their number, and the height of their supply the conditions of their nucleation quite clearly change. For example, Fig. 2 shows the change in the number of nuclei created at the stage of dispensing drops of a size $d_d = 4.9 \text{ mm}$, depending on the height h , for a different number of supplied drops n_d .

From Fig. 2 it follows that in the case of dispensing liquid just above the bed ($h = 0.02\text{m}$), the number of resulting nuclei is similar to the number of supplied liquid drops, which means that every drop approximately seeks to create a single germ. With the increase of height h there follows a significant increase in the number of resulting nuclei, which means that the supplied drops are prone to break-up, which results in a greater number of nuclei. The dependence of number of created nuclei to the specified number of drops ratio defined with a coefficient $\zeta = n_n/n_d$, on the height h for different sizes of droplets is compared in Fig. 3.

Figure 3 shows that the coefficient ζ considerably increases with the increase of droplet size, and for $d_d = 3.0$ take values close to 1. This means that in case of this droplet size no break-up occurs and the number of nuclei is virtually equal to the number of given drops.

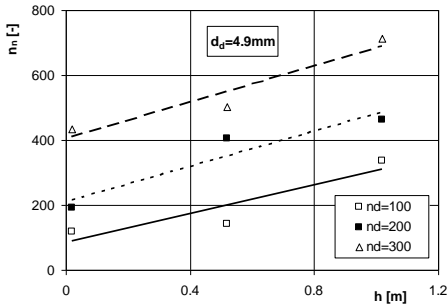


Fig. 2. Effect of liquid droplets supply height on the number of resulting nuclei

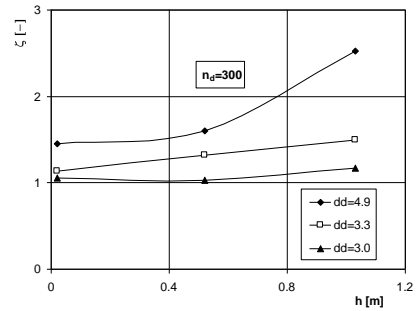


Fig. 3. Dependence of coefficient ζ on the height h , for different sizes of droplets d_d

Obtained dependencies indicate that the behavior of drops in a powder bed determines both its size (mass) and the speed with which it hits the bed. The speed of a drop hitting the material u was calculated based on correlation proposed by Range and Feuillebois (1998):

$$u = \sqrt{\frac{g}{A}(1 - e^{-2Ah})}, \quad (1)$$

$$\text{with } A = \frac{3c_f \cdot \rho_a}{4\rho_d \cdot d_d},$$

where $c_f = 0.796$ is the friction coefficient for a drop falling in air

g - gravitational acceleration, m/s^2

h - height of the needle tip above the bed, m

ρ_a - air density, kg/m^3

ρ_d - drop density (distilled water), kg/m^3

d_d - drop diameter, m .

Taking into account the weight of a falling drop m_d and its speed at impact u the kinetic energy of the drop was determined from the formula:

$$E_d = 0.5m_d \cdot u^2 . \tag{2}$$

Calculated speeds and kinetic energy of drops at the moment of impact with the powder are summarized in Table. 2.

The effect of drop kinetic energy on the value of coefficient ζ , with a fixed number of added drops n_d is shown in Fig. 4.

From Fig. 4 it follows that the effect of liquid supply height and drop size on their degree of decomposition, expressed with coefficient ζ , may be well defined with the kinetic energy of droplets colliding with the powder. A certain influence of the total number of droplets of liquid supplied to the bed may be observed here. The higher the value n_d coefficient ζ decreases. This can be elucidated by the fact that with a greater number of drops supplied to the bed a greater probability of their coalescence exists which influences a smaller number of obtained nuclei. An analogous relationship, shown in Fig. 5, was also obtained for a smaller intensity of binding liquid drop dispensing $Q_w = 0.025 \text{ cm}^3/\text{s}$.

Table 2. Drop impact velocity and energy

Drop diameter d_d [m]	High of the needle tip above the bed h [m]	Drop impact velocity u [m/s]	Drop mass m_d [kg]	Drop impact energy E_d [J]
$2.4 \cdot 10^{-3}$	0.02	0.625	$7.24 \cdot 10^{-6}$	$1.412 \cdot 10^{-6}$
	0.52	2.962		$3.174 \cdot 10^{-5}$
	1.02	3.872		$5.425 \cdot 10^{-5}$
$3.0 \cdot 10^{-3}$	0.02	0.625	$1.41 \cdot 10^{-5}$	$2.761 \cdot 10^{-6}$
	0.52	3.006		$6.386 \cdot 10^{-5}$
	1.02	3.980		$11.198 \cdot 10^{-5}$
$3.3 \cdot 10^{-3}$	0.02	0.625	$1.88 \cdot 10^{-5}$	$3.676 \cdot 10^{-6}$
	0.52	3.022		$8.592 \cdot 10^{-5}$
	1.02	4.021		$15.212 \cdot 10^{-5}$
$4.9 \cdot 10^{-3}$	0.02	0.626	$6.16 \cdot 10^{-5}$	$1.205 \cdot 10^{-5}$
	0.52	3.076		$29.151 \cdot 10^{-5}$
	1.02	4.160		$53.297 \cdot 10^{-5}$

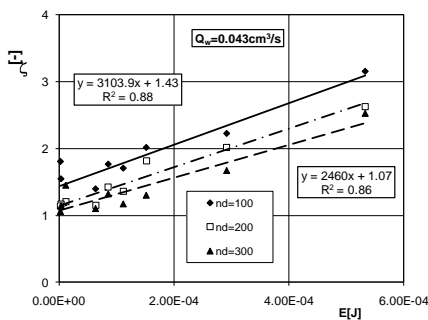


Fig. 4. Dependence of coefficient ζ on the kinetic energy of drops falling on the bed

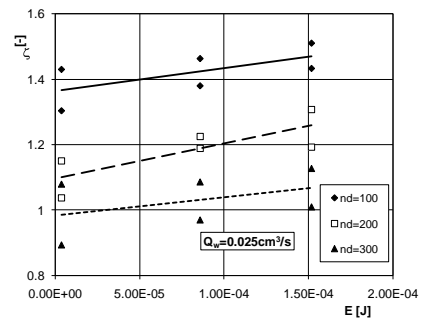


Fig. 5. Dependence of the coefficient ζ on the kinetic energy of drops falling on the bed

One may conclude about the mechanisms of formation and growth of nuclei at the stage of supply of binding liquid droplets to bed based on the size of nuclei created. When every drop strives to create a single germ, their dimensions are a reflection of the size of dispensed droplets. When it comes to breaking up of droplets, there can be created a greater number of droplets of smaller size. During the movement of wet powder bed, other mechanisms influencing the number and size of nuclei created, i.e. coalescence, wear, layering may occur. The importance of individual mechanisms may depend on the conditions for the supply of liquid to the bed and change during the process.

For example, Fig. 6 shows the comparison of curves of quantitative size composition for different heights of liquid supply at a specified size and number of drops, and Fig. 7 compares mass-distribution curves at the same conditions of drops supply.

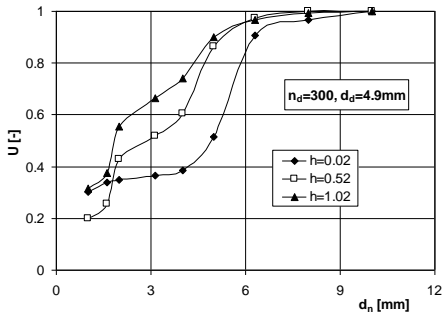


Fig. 6. Comparison of quantity-size distributions of nuclei

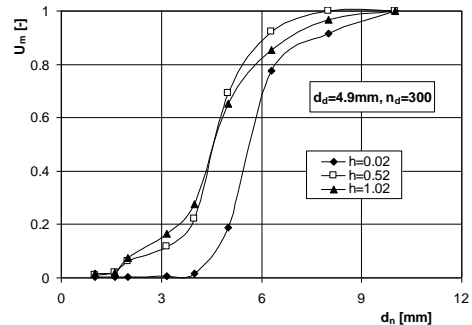


Fig. 7. Comparison of mass-size distributions of nuclei

In both graphs presented similar relationships can be observed. The increase in the amount of droplet supply results in increased quantitative and mass fraction of nuclei of a smaller size, which confirms previously observed trends regarding the conditions for liquid droplets break-up.

Figure 8 shows the comparison of the impact of liquid droplets supply on the average size of nuclei for the two droplet sizes, with a fixed number of them. For the droplet size $d_d = 4.9$ mm the increase of height h causes a reduction in the average quantitative size of nuclei, while for droplets of size $d_d = 3.0$ mm this impact was very slight. Figure 8 shows the comparison of changes in the average size of nuclei after 10 minutes of further granulation from the end of dispensing liquid.

It was found that during 10 min of granulation after the supply of liquids, the drop of germ size occurs. This may be caused by the destruction of even very weak agglomerates and minor thickening of their internal structure. The nuclei mass fraction in the total feed mass is very slight yet and nuclei are virtually suspended in the powder material which hinders their contact and mutual interaction. During this period, the liquid is no longer fed, and so there is no driving force for further formation and growth of nuclei and destructive mechanisms may play a greater role.

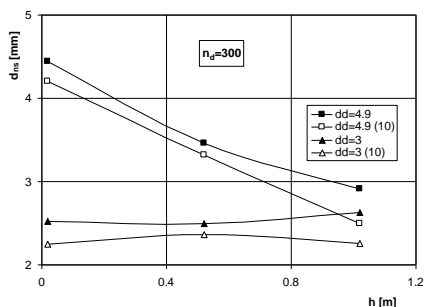


Fig. 8. Effect of liquid supply height on the average size of nuclei

5. Conclusions

On the basis of the obtained results the following conclusions were drawn.

1. Parameters of binding liquid droplets supplied to the powder material bed significantly affect the conditions of formation and growth of granule nuclei.
2. Effect of droplet size and their supply height above the bed on the number of resulting nuclei may be described with the kinetic energy of drops falling on the bed.
3. The increase of droplet supply height results in increased quantitative and mass fraction of nuclei of smaller size.
4. For larger droplets ($d_d = 4.9$ mm) the increase in the liquid supply height brings about reduction in the average quantitative size of nuclei while for smaller droplets ($d_d = 3.0$ mm) the effect is negligible.

Acknowledgments

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