

Introduction

Granulation, also described as agglomeration, pelletisation or balling is a process of the cohesion of fine particles such as dust or powders into larger aggregates (granules) with a defined mechanical strength in which the initial (starting) material grains may be still discerned. The agglomeration of fine particles of the solid body into larger clusters occurs as a result of physical and chemical mechanisms of binding.

In the process of wet granulation, the particles subjected to mixing (in tumbling drums, discs, fluid bed, fast-cutting mixers, etc.) are sprayed with binding liquid that binds the grains by means of a combination of capillary and viscosity forces. Stronger intragranular bonds may form upon further operations such as drying, sintering.

Granulation has a broad scope of application in numerous industries such as mineral processing, agriculture, detergent chemistry, pharmaceutical industry, food processing as well as chemical agents. In the chemical industry it is estimated for as many as 60% of the products to be manufactured as powder or dust and 20% contain additives in these forms. Granulation is a key stage in the processing or manufacture of products in many industries. Inadequate performance of granulation, yielding a product with undesired parameters may cause grave problems at successive stages of processing (agglomeration, segregation, low efficacy during tableting) as well as obstruct the application of the finished product.

One of the commonly applied modes of agglomeration is non-pressure agglomerative granulation performed in rotational discs with a tumbling motion of the filling. The method is attractive in terms of cost-effectiveness in light of low investment and exploitation expenses. For most materials, the processing of powder or dust into granulate requires delivering the adequate volume of damping or binding liquid to a granulated filling. Of fundamental importance for the course of the granulation process and the properties of the finished product are the phenomena and metamorphoses occurring at the border between the phases of media participating in the tumbling motion of the dampened material bed.

The mechanism responsible for the nucleus formation and agglomerate growth depend on the media used in the process as well as the construction parameters of the device applied and the conditions of the process [1 ÷ 5].

The studies on the conditions of disc granulation presented in the literature available concern, inter alia, the kinetics of the process [6, 7] as well as the issues associated with the circulation of the granulated filling in the disc and its dynamics [8 ÷ 9]. The said works, however, mainly deal with tests conducted in a periodical system.

Continuous disc granulation has a broad scope of application for industrial purposes but is rarely subject to laboratory studies. This is the reason behind few publications concerning research in this field [10 ÷ 12].

The results of the tests concerning continuous disc granulation performed for the purpose of our research shall enable better understanding of the effect of selected parameters of the continuous process on the course of granulation and the properties of the finished product.

Aim of the study

The aim of the study was to assess the effect of selected process and equipment parameters: intensity of fine-grained material delivery to the granulator, disc axis inclination angle as well as the rotational speed of the granulator in the course of the continuous disc granulation process as well as the properties of the finished product.

Materials, equipment and methodology for study

Foundry bentonite obtained from the Works "ZĘBIEC" served as the model fine-grained material. It had the following parameters:

- grain size: $0 \div 0.16\text{mm}$
- mean grain size $d=0,056\text{mm}$
- bulk density $\rho_m = 789.5 \text{ kg/m}^3$
- density $\rho = 2,420 \text{ kg/m}^3$.

Distilled water was employed to dampen the bed during the process.

The study of the granulation was performed on the equipment depicted in Figure 1.

The motoreducer (1) by means of a belt transmission drove the shaft attached to the granulator disc (2). The disc rotational speed was controlled by means of an inverter (3) and determined with a tachometer.

The damping liquid (water) was delivered drop-wise onto the moving granular bed situated on the disc from a tank (4), by means of a hydraulic system ended with a

sprinkler (6). A constant intensity of the liquid delivery was determined by means of a rotometer (5). In order to secure uniform damping conditions with variable intensity of damping liquid delivery (droplets of identical size), a varying number of sprinkler outlet nozzles were used (2, 3 and 4), proportionately to the intensity of fine-grained material delivery (raw material).

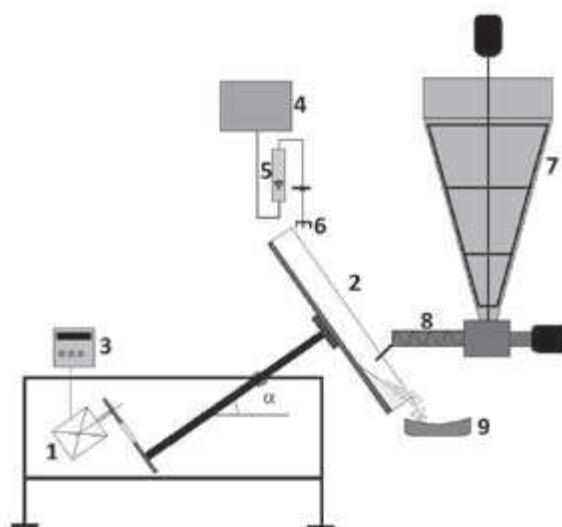


Fig. 1. Schematic diagram of the equipment
1. motoreducer, 2. granulator disc, 3. inverter, 4. binding liquid tank, 5. rotometer, 6. sprinkler, 7. powder feeder tank, 8. feeding screw, 9. product container

Fine-grained material was delivered to the granulator by means of a powder feeder DSK-10p, manufactured by Machine Construction Works "Hydrapress" S.A. The device ensured fluent control of the mass capacity of the powder delivered within the range of up to 150 kg/h. The feeder comprised a tank (7) in which the raw material was mixed by means of the in-built stirrer and a feeding screw (8) with a continuous regulation of rotational speed.

The finished product (wet granulate), tumbling over the rim of the disc, was collected in a container (9).

Preliminary studies were carried out with a view to determining the conditions of research performance and selecting the range of variations in the parameter applied. As a result, the following values of variable parameters were accepted:

- inclination angle of disc axis to the horizontal plane $\alpha=37^\circ$, 45° and 53°
- disc rotational speed $n=(0.15 \div 0.30)n_{kr}$.

where the critical speed equals:

$$n_{kr} = \frac{0.705}{\sqrt{D}} \sqrt{\sin(90-\alpha)} \quad (1)$$

D – inner disc diameter, m

- intensity of the delivery of powder material to the granulator $Q=0.136 \div 0.272$ kg/min

Rotational speeds applied for respective disc inclination angles are shown in Table 1.

Table 1

Disc rotational speed

Sg.	Disc inclination angle α [°]	Critical speed n_{kr} [obr/min]	Speed to critical speed ratio n/n_{kr}	Disc rotational speed n [obr/min]
1	37°	53.5	0.15	8
2			0.2	10.7
3			0.3	16
4	45°	50.3	0.15	7.5
5			0.2	10
6			0.3	15
7	53°	46.5	0.15	7
8			0.2	9.3
9			0.3	13.9

The study of continuous disc granulation were conducted in accordance with the following constant values of parameters:

- disc diameter $D = 0.5$ m
- disc rim height $H = 0.1$ m
- ratio of the intensity of damping liquid delivery to the intensity of powder material delivery $Q_w/Q=0.4$
- site of the delivery of fine-grained material and binding liquid.

Variable process parameters applied in individual trials are show in Table 2.

Table 2

The parameters used during study

Sample no.	Inclination angle α [°]	Material feeding intensity Q [g/min]	Speed to critical speed ratio n/n_{kr}	
1	45°	272	0.2	
2			0.3	
3			0.15	
4		136	204	0.3
5				0.2
6				0.15
7		204	272	0.15
8				0.2
9				0.3
10	37°	204	0.2	
11			0.3	
12			0.15	
13		136	272	0.15
14				0.2
15				0.3
16		272	204	0.2
17				0.3
18				0.15
19	53°	204	0.3	
20			0.15	
21			0.2	
22		272	136	0.3
23				0.15
24				0.2
25		136	204	0.3
26				0.15
27				0.2

At the outset of each trial a mass of powder material predetermined upon preliminary studies, equivalent to the amount of filling that allowed rotary motion without raw material spill from the disc, was delivered to the disc. Subsequently, the granulator drive was turned on and water was fed onto the tumbling bed at a preset rotational speed of the disc. The dampening of the filling at this stage lasted until the water content constituted 40% of the powder material mass. Upon the achievement of the desired moisture of the filling, the feeder of fine-grained material was activated. At this stage both the binding liquid and the bulk material were delivered, while maintaining a steady ratio of the liquid mass to the mass of the raw material equal to 0.4.

In the period of preliminary damping, the granulated filling lost some of its volume as a result of granule structure densification. In effect, despite continuous delivery of powder material and liquid, the granulate started to tumble over the rim of the disc not immediately but after some time. At particular intervals samples of the product tumbling over the disc were collected for 1 minute. The process was run until consecutive samples did not show significant differences in regard to mass and granular composition.

Each sample, once dampened, was divided into fractions on a set of screens with the eyelet sizes of: 0.4, 1, 2, 3, 4, 5, 6.3, 8, 10 and 12.5mm. Individual fractions were weighed by means of laboratory scales with the accuracy of 0.01 g and, subsequently, dried in a laboratory dryer at the temperature 95°C for 24h. On the basis of the balance between the initial and final mass upon drying, the moisture of respective fractions was determined for a given product sample. The screen analysis was also performed with respect to the granulated filling remaining on the disc upon the termination of the process.

Results

On the basis of the results obtained during the studies, the effect of the parameters investigated in the course of continuous granulation and the properties of the finished product were analysed.

Figures 2÷4 demonstrate exemplary size distribution of the product samples collected after various durations of the continuous process since the commencement of the continuous delivery of fine-grained material, at various values of process and equipment parameters.

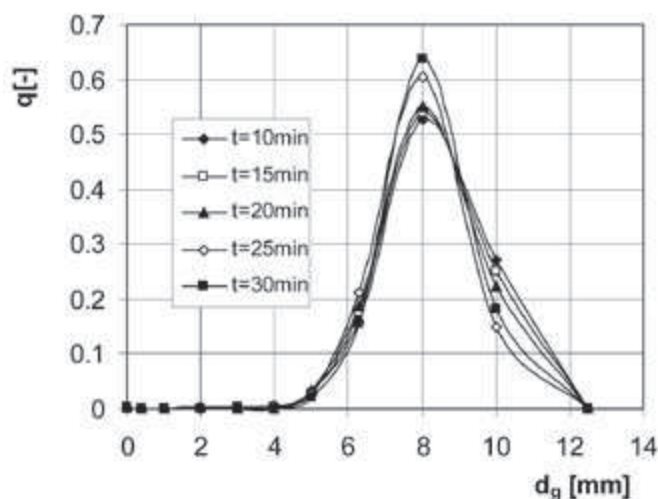


Fig. 2. The comparison of size distribution of the product for different process length times ($\alpha=45^\circ, n=0,3n_r, Q=272 \text{ g/min}$)

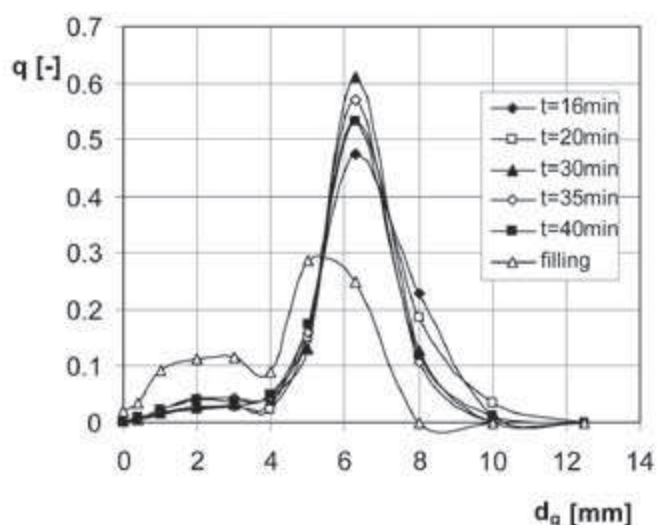


Fig. 3. The comparison of size distribution of the product for different process length times ($\alpha=37^\circ, n=0,2n_r, Q=136 \text{ g/min}$)

Figures 3 and 4 depict an additional comparative analysis of the size distribution of the product obtained after a particular process duration as well as the filling remaining in the disc upon the collection of the last sample.

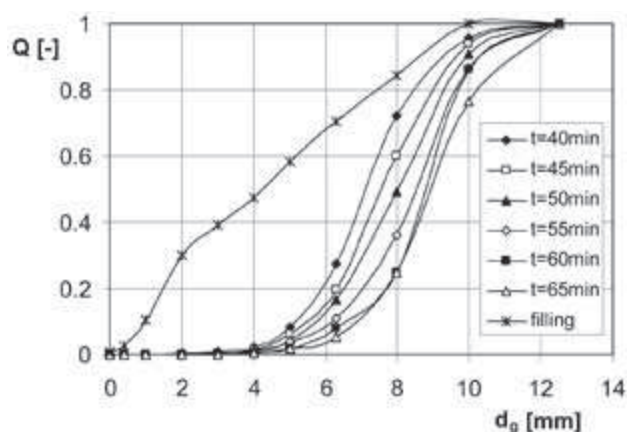


Fig. 4. The comparison of cumulative size distribution of the product for different process length times ($\alpha=53^\circ, n=0,15n_r, Q=136 \text{ g/min}$)

What may be inferred from figures 3 and 4 is that the size distribution of the bed that remains in the granulator upon the termination of the process clearly differs from the granular composition of the product that tumbles over the disc during the continuous process. The bed predominantly contains particles that are much finer than the granules tumbling over the disc during the continuous process and some non-granulated fragments of the raw material. This testifies an accurate segregation of the particles in the granulated bed that cause granules of an adequate size to tumble over the disc.

Upon the analysis of water content in respective fractions of granules, it was found that their moisture w , defined as the ratio of their water content to the mass of dry agglomerates tends to rise significantly along with the particle diameter. The comparative analysis of granule moisture in respective fractions for various process durations is shown in Figure 5.

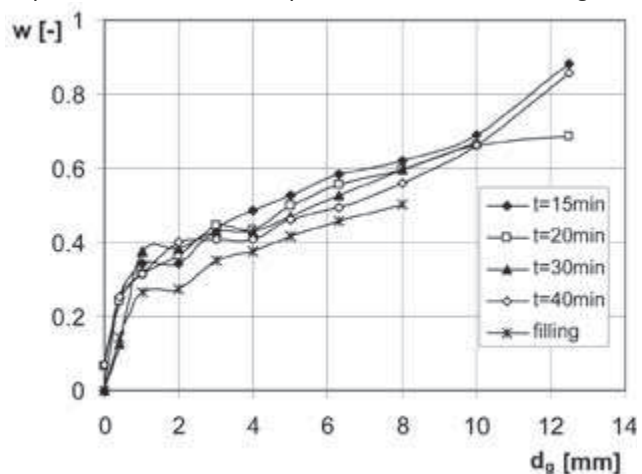


Fig. 5. The comparison of water content (moisture w) in individual size fractions of granulated product ($\alpha=45^\circ, n/n_r=0,15, Q=204 \text{ g/min}$)

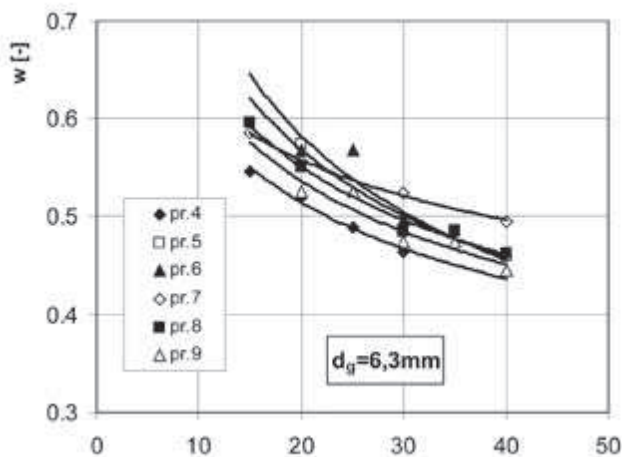


Fig. 6. The change in moisture content during granulation for size fraction $d_g = 6,3\text{mm}$

It was established that the moisture content in the granules in individual granular fractions declined in time and at the final stage of the process it reaches stability at a certain level. Water content variation in the granules throughout the process for a particular fraction has been presented in Figure 6.

For the purpose of describing granular composition of the granulate obtained in a particular process time the following statistical moments have been applied:

zero moment of first order (mean particle size)

$$d_{gs} = m_1 = \sum_{i=1}^n x_i \cdot d_{gsi} \quad (2)$$

where:

x_i – mass content of grains of i-th class

d_{gsi} – mean size of grains of i-th class

central moment of secondary order (distribution variance)

$$\sigma^2 = \sum_{i=1}^n x_i \cdot (d_{gsi} - d_{gs})^2 \quad (3)$$

coefficient of variation – the relation of the standard deviation to the mean grain size

$$\frac{\sigma}{d_{gs}} \quad (4)$$

Exemplary variations in mean granule size in samples collected after a particular duration of the continuous process have been presented in Fig. 7, while the variation in coefficient of variation – in Figure 8.

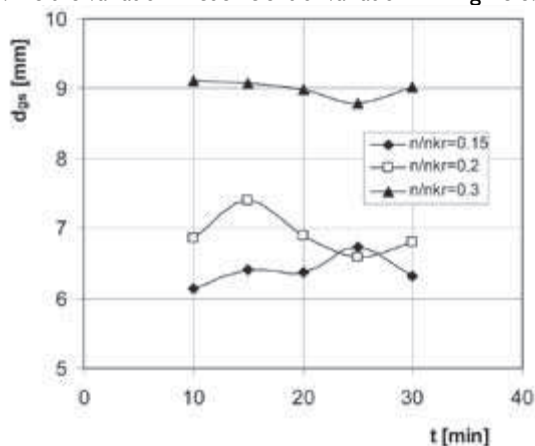


Fig. 7. The change in the average size of the granules in time ($\alpha=45^\circ$, $Q=272$ g/min)

It may be inferred from Figure 7 that the modification in the mean granule size during the process (in successive collected samples) at set process parameters is minor with the corollary that a continuous process promptly becomes stable. The slight change in the coefficients of variation of the size distribution for samples collected at successive intervals, as detailed in Figure 8, further supports this claim.

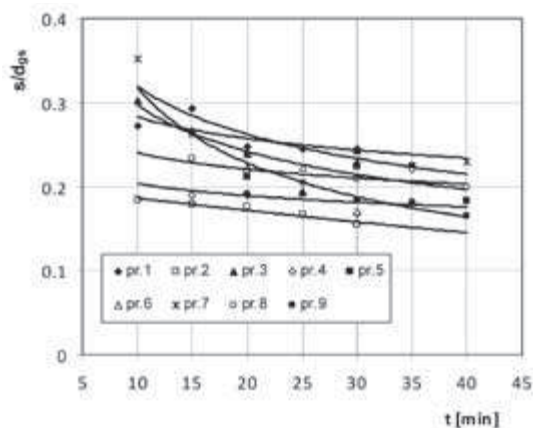


Fig. 8. The change in variation coefficient during the process ($\alpha=45^\circ$)

Significant differences in granule size are caused by the alterations in the process parameters applied during studies, leading to significant variation in the drum filling throughout the process and the concomitant mean residence time of the filling in the granulator.

The mean residence time of the material on the disc was determined in accordance with the following correlation:

$$\tau = \frac{M}{Q + Q_w} \quad (5)$$

where: M – mass of material remaining on the disc at the end of the process, kg

The effect of the mean residence time of the material inside the granulator on the mean granule size has been depicted in Figure 9.

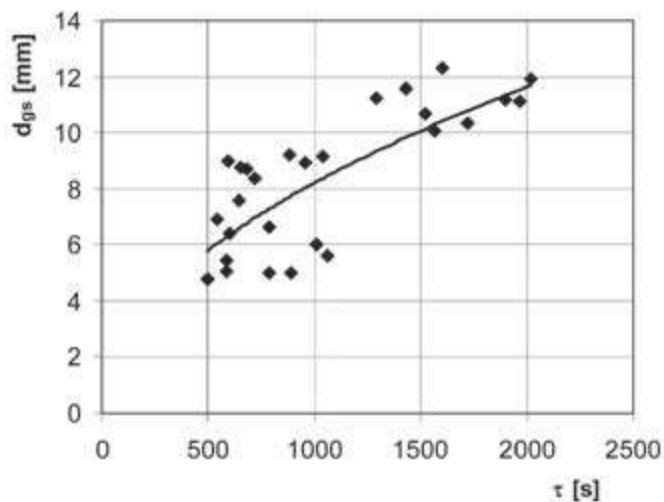


Fig. 9. The effect of the mean residence time on the mean size of granules

Basing on the set of all experiential points provided in Figure 9, a general tendency may be seen concerning the mean granule size becoming greater relative to the residence time of the bed in the granulator. However, individual points form specific groups associated with the angle of the disc inclination that is the main factor affecting the mass of the bed tumbling in the granulator. The interrelations between residence time and granule size with respect to three inclination angles of the disc have been detailed in Figure 10.

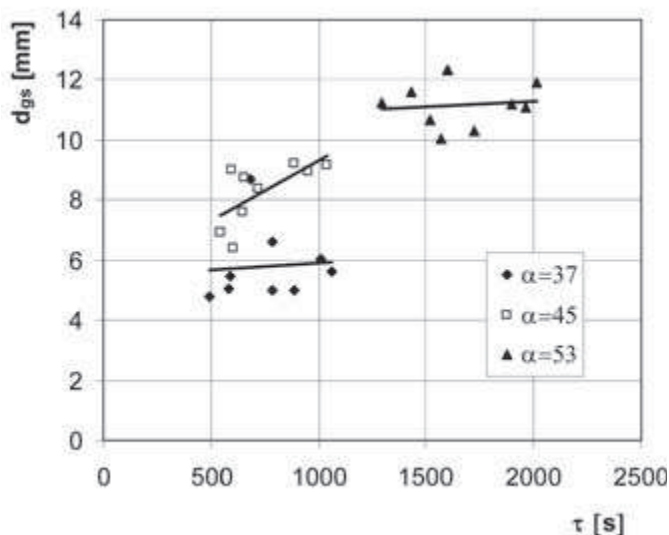


Fig. 10. The effect of the mean residence time on the mean size of granules for different angles of disc inclination α

In order to determine the effect of the variable study process parameters on the value of mean granule size in the finished product (upon the establishment of the conditions of the continuous process), the following power model has been employed:

$$d_{gs} = A \cdot \alpha^b \cdot Q^c \cdot \frac{n}{n_{kr}}^d \quad (6)$$

where:

A - constant,

b, c, d – power exponents

As a result of power regression of a number of variables, the following equation has been derived:

$$d_{gs} = 0,0252 \cdot \alpha^{1,86} \cdot Q^{-0,185} \cdot \frac{n}{n_{kr}}^{0,2} \quad (7)$$

with the correlation coefficient $R^2=0.88$

The comparative analysis of mean granule size as calculated on the basis of equation (7) with experiential values derived from the size distribution of the finished product has been provided in Figure 11.

The values of the power exponents in the equation derived proves that the most significant effect on the mean particle size in the product obtained during continuous disc granulation process is exerted by the inclination angle of the granulator disc axis. A positive value of the power exponent accompanied by this parameter indicates that as the value of the angle α increases, the mean granule size d_{gs} grows considerably. This is due to both the increase in the mean residence time of the bed on the disc as well as greater dynamics in the tumbling bed, consequent upon greater mass and the concomitant greater height of the granulated filling.

The effect of other examined parameters proves minor. The rise in the intensity of powder material delivery to the granulator has a slightly reductive influence on the mean granule size as a result of the decrease in the mean residence time of the granulated bed on the disc. The augmentation of the rotational speed of the disc causes the mean granulate particle size to grow as a consequence of more intense bed motion on the disc, affecting the frequency and strength of the interactions between the tumbling particles of the bed, which is inductive to their aggregation.

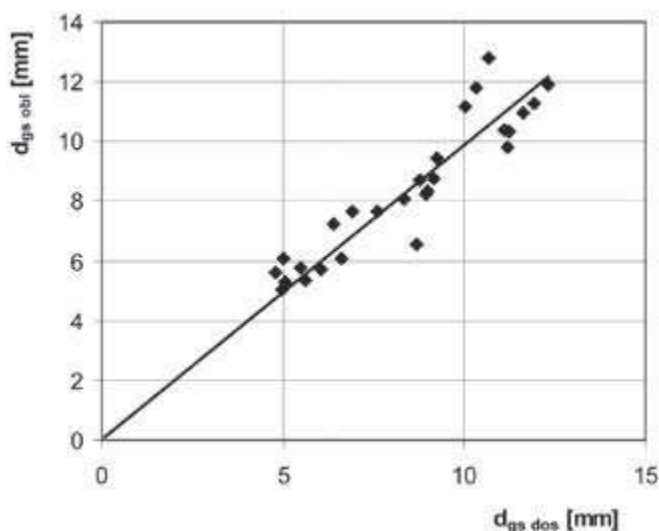


Fig. 11. The comparison of calculated and experimental values of the average size of granules

Conclusions

On the basis of the studies conducted with respect to continuous disc granulation, the following conclusions may be drawn:

- disc inclination angle exerts the greatest influence on the properties of the granulation product; the augmentation of inclination angle of disc axis to horizontal position causes an increase in the mean size of forming granules
- increase in the intensity of powder raw material delivery to the granulator leads to a minor decrease in the size of forming particles
- increase in the rotational speed of the disc gives rise to the increase in the mean granule size
- granules characterised by greater size have a larger content of the binding liquid
- the prolongation of the residence time of the granulated material on the disc triggers the increment in the size of forming granules
- as the residence time of the material in the granulator increases, the product acquires greater homogeneity
- the inclination angle of disc axis is the major factor that affects the residence time of the material in the granulator .

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