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THE EFFECT OF WETTING CONDITIONS ON THE STRENGTH OF GRANULES

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Results of investigations of the effect of the size of wetting liquid droplets and particle size distribution of a fine-grained raw material on the mechanical strength of granules formed during wet drum granulation were discussed. The process of granulation was carried out batch-wise in a drum granulator 0.5 m in diameter and 0.4 m long at rotational speed $0.33s^{-1}$ and constant volumetric drum filling degree $\varphi = 0.1$. On the bed tumbling in the drum the wetting liquid (distilled water) was supplied at a constant flow rate $Q_w = 12 \cdot 10^{-3}$ m^3/h. The size of wetting droplets was changed using various air flow rates through pneumatic spray nozzles in the range Q_a=1.0 to 3.0 m^3/h and applying a sprinkler which supplied /drop-wise/ the liquid uniformly along the entire drum length. In the whole experimental cycle constant mean saturation degree of the feed equal to S = 0.293 was used. The effect of wetting droplet size and particle size distribution of the raw material on the breaking force and corresponding breaking stress was discussed.

Key words: granulation, agglomeration, size distributions, strength of agglomerates

INTRODUCTION

In wet granulation the main role is played by phenomena which take place at the liquid-solid-gas interface that depend on the properties of media involved in the process and also on process conditions. For these reasons the choice of wetting conditions for a fine-grained bed of specified physical properties constitutes one of main problems of every granulation process.

One of the often applied agglomeration methods is pressure-free granulation carried out in rotary drums. During the tumbling of a wetted fine-grained bed, solid particles interact with liquid droplets and air. The size and type of forces acting on single material particles and their agglomerates strictly depend on the properties of particular media, their relationships and in particular on the particle size distribution of

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the raw material, the shape of particles and extent of spraying of the wetting liquid. The effect of the quantity and properties of wetting liquid on the mechanisms of agglomerate growth and their properties was studied by many researchers (Kapur and Fuerstenau 1969, Sastry and Fuerstenau 1973, Gluba et al. 1990, Iveson and Litster 1998). These studies carried out for various solid-wetting liquid systems, confirmed a significant impact of the amount and properties of the liquid on growth kinetics of agglomerates and their physico-mechanical properties. There are a few references in the literature to the influence of the wetting liquid droplet size on granulation effects.

Mechanical strength of agglomerates is one of the main features determining their further applicability or processing. There are many methods of defining and measuring the strength. Depending on needs, impact, wear, compression, bend and tensile tests are used (Błasiński and Gluba 1981, Schubert 1975, Kristensen et al. 1985, Gluba and Antkowiak 1988) In all these cases strength is determined by means of testing machines, so the theory and transfer of results of these measurements to other stresses are more or less hampered. Strength tests are, however, a significant source of information on the quality and structure of granulated product.

Production of a granular material with defined properties (mechanical strength) from a fine-grained raw material requires an appropriate method of granulation and selection of proper process parameters. One of little known problems is the determination of the effect of wetting liquid droplet size and physical properties of the material being granulated, and particle size distribution in particular, on the strength of granules being formed. This paper tries to explain the above mentioned relations.

AIM OF THE STUDY

The aim of the study was to determine the effect of the size of wetting liquid droplets and particle size distribution of a fine-grained raw material on the mechanical strength of granules formed during wet drum granulation.

EXPERIMENTAL

MATERIALS

Fine-grained dolomite flour was used in the experiments. Raw materials for testing were composed of five size fractions of the dolomite flour with particle size ranging to 10 μm, to 15 μm, to 60 μm, to 100 μm and to 250 μm. Particle size distribution of each fraction was determined using a laser particle size analyser “Analysette 22”. The particle size distribution was described by the statistical moments: mean particle size d_m, variance of dimensions s^2, asymmetry coefficient γ_1 and concentration coefficient γ_2. On the basis of data obtained from the laser analyser particle size distributions of raw materials to be granulated were calculated numerically (by mixing input fractions) assuming that they were geometrically similar. In the calculations the following were assumed constant: variation coefficient: $s/d_m = 1.07$ and asymmetry coefficient $\gamma_1 =$

2.00 for different values of mean particle size d_m ranging from 10.6 to 28 μm. Grain-size compositions of mixtures obtained (denoted by symbols D1 to D5), which are raw materials for the granulation process, are shown in Fig. 1. Such a selection of particle size distributions enables an evaluation of the impact of mean particle diameter on the granulation process and the mechanical strength of granules.

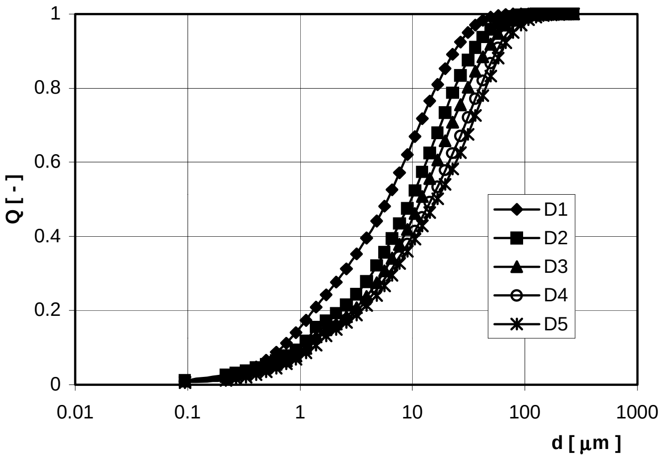

Fig. 1. Comparison of particle size distributions of raw materials

EXPERIMENTAL RIG AND METHODS

The process of granulation was carried out batch-wise in a drum granulator 0.5 m in diameter and 0.4 m long at rotational speed $0.33s^{-1}$ and constant volumetric drum filling degree $\varphi = 0.1$. The feed volume was determined each time on the basis of mean bulk density of particular raw materials. On the bed tumbling in the drum the wetting liquid (distilled water) was supplied at a constant flow rate $Q_w = 12\cdot10^{-3}$ m^3/h. The size of wetting droplets was changed using various air flow rates through pneumatic spray nozzles in the range Q_a=1.0 to 3.0 m^3/h and applying a sprinkler which supplied (drop-wise) the liquid uniformly along the entire drum length.

The size distribution of wetting liquid droplets supplied by a pneumatic nozzle was determined by a DANTEC laser analyser. The droplet size distribution was analysed at a distance of 100 mm from the nozzle outlet along the radius of dispersed liquid stream every 2.5 mm from the nozzle axis. A comparison of averaged droplet size distributions for the whole stream at specified nozzle operating parameters (q) is shown in Fig. 2, while the relation of mean droplet size d_{dm} in the stream and distribution range s/d_{dm} is illustrated in Fig. 3. From Fig. 3 it follows that with an increase of the liquid atomisation $q=Q_w/Q_a$ the mean droplet size in the stream increases, while the coefficient of distribution variability s/d_{dm} decreases which means that the stream becomes more homogeneous.

During drop-wise wetting the bed was wetted with droplets of the same size equal to about 3 mm. In the whole experimental cycle constant wetting was used which was determined by the mean saturation degree of the feed equal to S = 0.293.

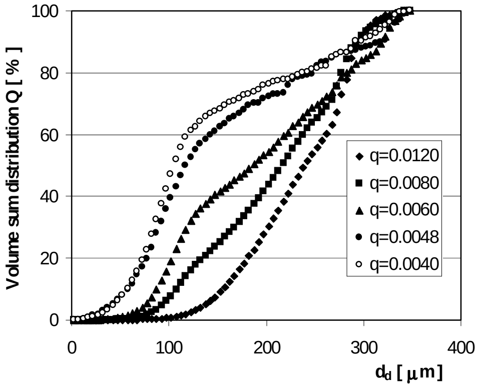

Fig. 2. Size distributions of wetting liquid

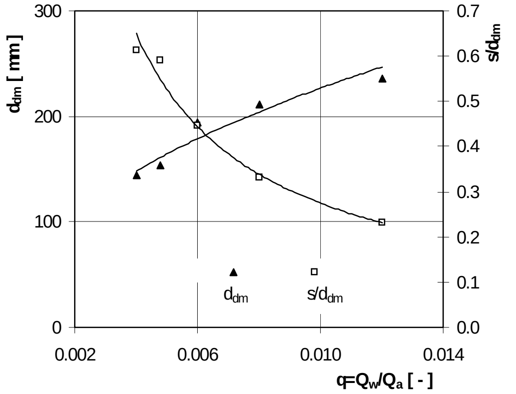

Fig. 3. Dependence of mean droplet size d_{dm} and droplets coefficient s/d_{dm} on the liquid dispersion q

After completing the wetting, the process of granulation was continued until the moment when the granulated material started to stick to the drum walls because water had been pressed off. In determined time intervals, samples representative of the whole feed were taken. On this basis properties of the product obtained were specified. The first sample was taken immediately after wetting (granulation time t = 0) and the last one in the moment when water pressed out to the granule surface made them stick to the inner surface of the drum.

In order to determine the compression resistance, a single dry granule from a size fraction (of given dimensions) was placed between two parallel plates of the strength testing machine, where it was loaded with an increasing compressive force until the sample was damaged. Schematic diagram of the equipment used for compressive strength tests is shown in Fig. 4.

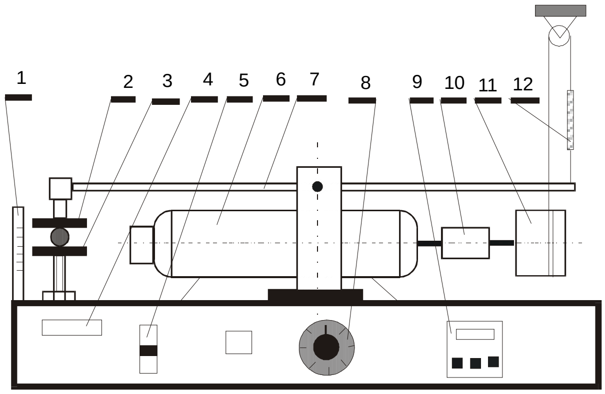

Fig. 4. Schematic diagram of the equipment for compressive strength tests: (1) scale, (2, 3) compressing plates, (4) feed control, (5) direction switch, (6) engine with a reducer, (7) double-arm lever, (8) potentiometer, (9) torque meter, (10) torque sensor, (11) winding reel, (12) spring

The compressive force was generated as a result of tension of the spring caused by winding of the flexible connector onto the reel, which rotated at a given rotational velocity. Constant rotational velocity of the drum was applied, which gave a constant growth rate of the loading force ΔN=0.245 N/s.

RESULTS

The value of breaking force was calculated according to eq. (1) on the basis of twisting moment on the reel shaft, recorded at the instant when the sample was destroyed:

$$P = \frac{M_s}{D/2} \tag{1}$$

The breaking force for granules from a given size fraction was assumed to be the arithmetic mean from values obtained for 10 single samples. Breaking compressive stresses were determined on the basis of mean value of breaking force and granule size according to the formula:

$$\sigma = \frac{4P}{\pi d_g^{\ 2}} \tag{2}$$

Figure 5a shows an example of the dependence of breaking force and Fig. 5b the effect of breaking stresses on the diameter of granules obtained from one material (D3) for different granulation times at determined mean wetting liquid droplet size (d_{dm}=0.144mm).

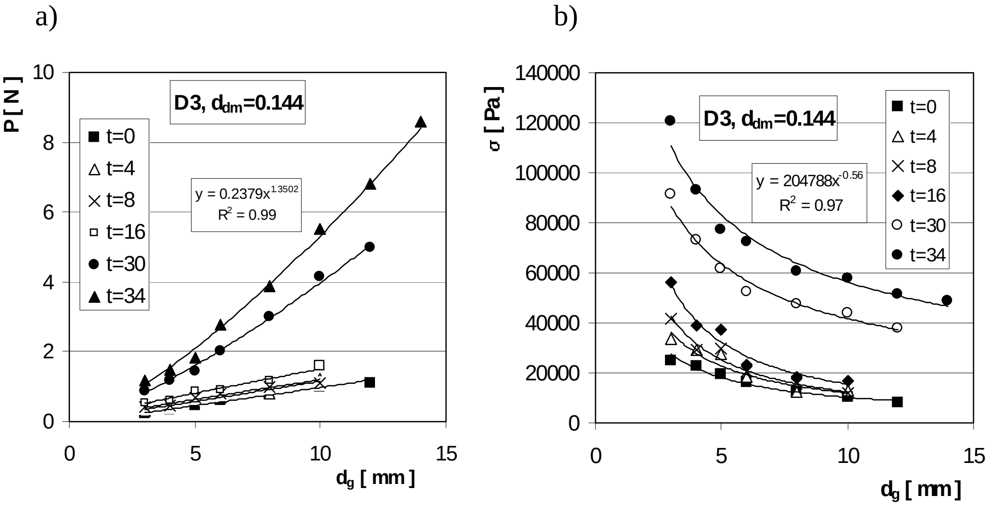

Fig. 5a, b. Dependence of breaking force (P) and breaking stresses (σ) on granule size

For all tested materials relations similar to those shown in Figs. 5a, b were obtained. In every case the growth of granulation time results in a stepwise increase of granule strength. The dependence of breaking force on granule diameter is approximated with high accuracy by the power function:

$$P = A \cdot d_g{}^{B} \tag{3}$$

while breaking stresses are determined by a function in the following form:

$$\sigma = A_1 \cdot d_g{}^{-B_1} \tag{4}$$

In order to estimate the effect of wetting liquid droplet size on strength of the produced dried granulated material, a comparison was made of curves P = f(d_g) and σ = f(d_g) obtained for a given raw material at the same granulation time. A character of the relations obtained for the granulated product shortly after completion of the wetting (t=1) is illustrated in Fig. 6a, b.

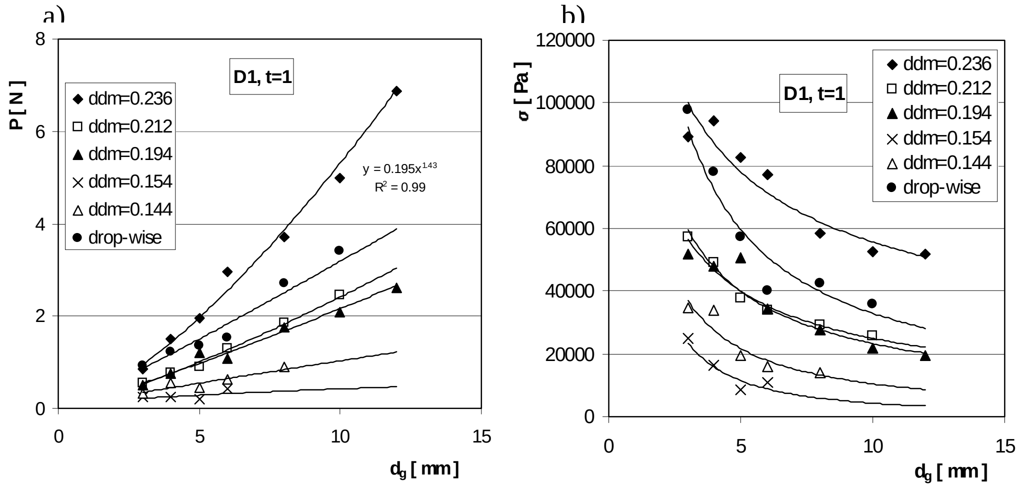

Fig. 6a, b. Dependence of breaking force (P) and breaking stresses (σ) on granule size

It is clear that the strength of product particles obtained in the initial period of granulation depends significantly on the wetting liquid droplet size. The highest strength have the granules formed when the wetting was carried out by a pneumatic nozzle at the smallest possible breaking of the liquid stream (the largest droplets). With an increase of the liquid distribution degree (a reduction of the droplet size) the compressive strength of granules gradually decreases. The strength of granules formed during the drop-wise wetting (droplets about 3 mm in diameter), in this initial granulation period is smaller than the values obtained for wetting with a nozzle using the largest droplets. It can be presumed that the size of droplets of the liquid supplied to the bed during the wetting affects the mechanisms of agglomerate formation, in particular the forces which bind material particles. Larger liquid droplets supplied to the fine-granular bed provide a possibility of occurrence of stronger liquid bridges which causes that the particles are closer to each other. Such a relation found for a determined, tested range of the wetting liquid droplet size cannot be probably extended onto arbitrary droplet sizes. At droplet sizes much exceeding the feed particle dimensions as used during the drop-wise wetting there are other mechanisms of agglomerate formation and growth. A large droplet, when falling down onto the fine-granular bed, spreads in the vicinity and forms a large nucleus, in which liquid occupies significant part of the interparticle space. What determines the position of particles in such a nucleus is the capillary negative pressure. The total number of droplets supplied to the bed during the drop-wise wetting is many times smaller than the number of liquid droplets during the wetting with a pneumatic nozzle and the droplets are of the same size. After completing the wetting process a more homogeneous product of larger granules is obtained. During further granulation, in the tumbling bed complex mechanisms are observed whose result is both the growth of agglomerates and a systematic concentration of their structure. As a consequence, water is pressed onto the agglomerate surface which causes an abrupt growth of the agglomerates due to combining of granules that earlier were separate, and in a very short time hamper significantly the further process because of an intensive sticking to the drum walls. The rate of these phenomena depends greatly on the liquid droplet size during wetting.

The strength of final product particles (corresponding to the surface overwetting) for particular raw materials also depends on the size of wetting liquid droplets. Examples of the relations $\sigma = f(d_g)$ for the final product obtained from one of the raw materials at different sizes of the wetting liquid droplets are shown in Fig. 7.

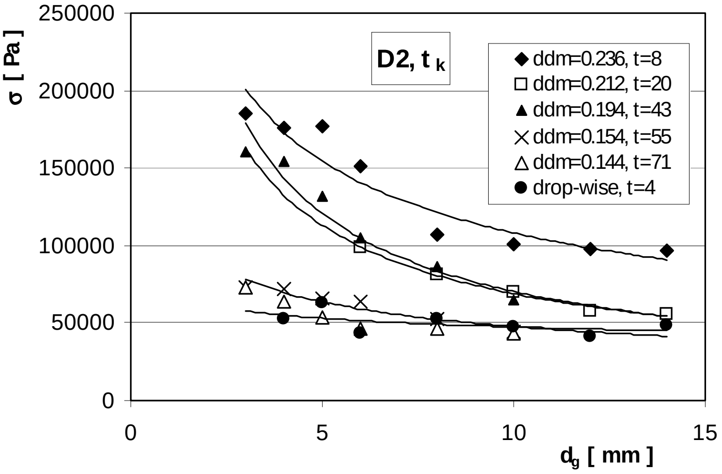

Fig. 7. Dependence of breaking stresses on granule size

Despite a relatively short time of granulation, of the highest strength are the agglomerates produced during wetting with the largest droplets supplied by the pneumatic nozzles. The lowest compressive strength is characteristic for the agglomerates obtained during the drop-wise wetting, for which the time necessary to reach the state of surface overwetting is also the shortest. This means that in this case the state of granule inner structure formed after the wetting changes slightly during further granulation. Hence, also an increase of strength as compared to the wetting with sprayed liquid is insignificant.

To evaluate the effect of particle size distribution of a raw material on product particle strength, the curves $P = f(d_g)$ and $\sigma = f(d_g)$ were compared for granulated material produced from different raw materials (with different average particle diameters) at a steady size of the wetting liquid droplets and granulation time. Example of diagrams of these relations are shown in Fig. 8. The effect of the particle size distribution is most distinct in the initial period of granulation. In the tested range of particle size distribution, the compressive strength of granules decreases with an increase of the mean material particle size. With an increase of the granulation time differences in the strength of granules made of particular raw materials become smaller. The least distinct differences in the strength of granulated product obtained from raw materials with different particle size distributions were obtained during the drop-wise wetting. For the product obtained after several minutes of granulation these differences are practically negligible.

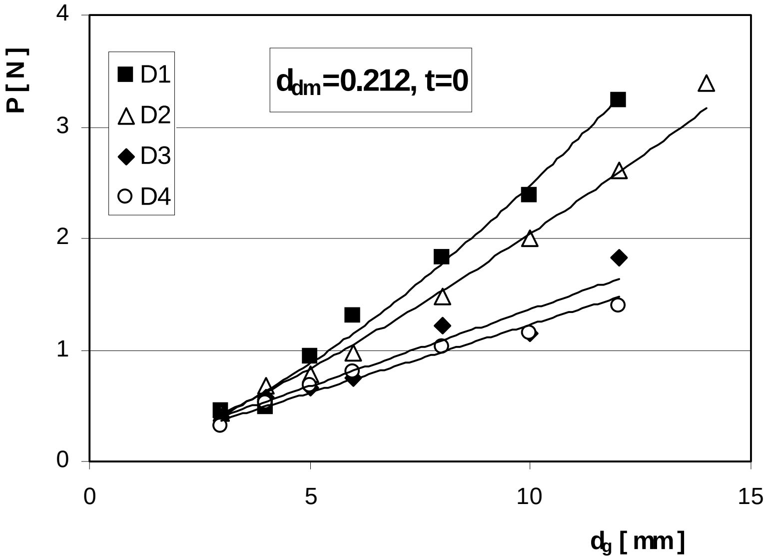

Fig. 8. Dependence of breaking force on granule size

CONCLUSIONS

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

1. Compressive strength of granules obtained during wet drum granulation depends strongly on the bed wetting parameters.
2. The size of droplets of the wetting liquid supplied to the bed during wetting has an influence on the granulation process, and as a result on the product properties including its mechanical strength.
3. An increase of the size of droplets supplied by pneumatic nozzles (in the tested range) leads to a product of higher strength.
4. During wetting with large droplets (the drop-wise wetting) the agglomerates grow very quickly, however, the obtained product is characterised by lower mechanical strength.
5. At the same size of the wetting liquid droplets a more resistant granulated product is obtained from the raw material with smaller mean particle diameters.
6. With an increase of the granulation time, there is a systematic increase of the strength of the obtained product particles.

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REFERENCES

BŁASIŃSKI H., GLUBA T., (1981), *Metody i aparaty do pomiaru wytrzymałości aglomeratów*, Inż. i Aparat. Chem., 4, 29-33

GLUBA T., ANTKOWIAK W., (1988), *Effect of wetting on granule abrasion resistance.* Aufbereitungs-Technik, 2, 76-80.

GLUBA T., HEIM A., KOCHANSKI B., (1990), *Application of the theory of moments in the estimation of powder granulation of different wettabilities*, Powder handling & processing, 2, 323-326.

IVESON S. M., LITSTER J. D., (1998), *Fundamental studies of granule consolidation. Part 2: Quantifying the effects of particle and binder properties,* Powder Technol., 99, 243-250.

KAPUR P.C., FUERSTENAU D.W., (1969), *A coalescence model for granulation*, I&EC Process Design and Development, 8, 56-62.

KRISTENSEN H. G., HOLM P., SCHAEFER T., (1985), *Mechanical Properties of Moist Agglomerates in Relation to Granulation Mechanisms*, Powder Technology, 44, 227-237

SASTRY K.V.S., FUERSTENAU D.W., (1973), *Mechanisms of Agglomerate Growth in Green Pelletization*, Powder Technology, 7, 97-105.

SCHUBERT H., (1975), *Tensile strength of agglomerates,* Powder Technology, 11, 107-119

NOMENCLATURE

d	-particle size of the raw material, μm,
D –	-winding reel diameter, m
d_g –	-granule diameter, mm
d_{gm}	-mean particle size of the granulated product, mm
d_d	-droplet size, mm
d_{dm}	-mean droplet size, mm
M_s –	-twisting moment during sample destruction, Nm
P –	-force causing granule breaking, N
$q=Q_w/Q_a$	-liquid dispersion
Q	-volume sum distribution
t	-granulation time after wetting, min,
σ –	-breaking stress, Pa

Gluba T., *Wpływ warunków nawilżania na wytrzymałość granulek*, Fizykochemiczne Problemy Mineralurgii 36, (2002) 233-242 (w jęz. ang.)

W pracy przedstawiono analizę wpływu stopnia rozbicia strumienia cieczy zwilżającej (wielkości kropel) oraz czasu prowadzenia procesu mokrej granulacji bębnowej na wytrzymałość mechaniczną wytworzonych cząstek granulatu ocenianą w stanie wysuszonym, przy użyciu testu na ściskanie. Jako materiał badawczy zastosowano mączkę dolomitową o zmiennym składzie ziarnowym. Skład granulometryczny surowca obliczano numerycznie przy założeniu stałości dwóch parametrów: współczynnika zmienności s/d_m = 1.07 oraz współczynnika asymetrii γ_1 = 2.00, dla pięciu wartości średniego wymiaru ziaren d_m w zakresie 10.6 do 28 μm. Proces granulacji prowadzono w sposób okresowy w granulatorze bębnowym o średnicy 0.5m i długości 0.4m przy stałej prędkości obrotowej 0,33 1/s i stałym objętościowym współczynniku wypełnienia bębna φ = 0.1. Na cyrkulujące w bębnie złoże podawano ciecz zwilżającą (wodę destylowaną) za pomocą dwóch dysz pneumatycznych przy stałym objętościowym natężeniu przepływu $Q_w = 12\cdot10^{-3}$ m^3/h. Wielkość kropel cieczy zmieniano stosując zmienne natężenia przepływu powietrza przez dysze w zakresie Q_a=1.0 do 3.0 m^3/h, oraz stosując zraszacz kroplowy, który podawał równomiernie na całej długości bębna ciecz w postaci kropel o wielkości ok. 3 mm. W całym cyklu badań stosowano stały współczynnik saturacji złoża to S = 0.293. Po zakończeniu nawilżania proces granulacji prowadzono do chwili gdy przewilżone na powierzchni granulki zaczęły przywierać do ścianek granulatora uniemożliwiając dalszy proces. Na podstawie próbek pobranych w określonych przedziałach czasowych określano właściwości wytrzymałościowe cząstek produktu.. Stwierdzono, że wielkość kropel cieczy zwilżającej istotnie wpływa na przebieg procesu granulacji, wielkość cząstek otrzymanego produktu i ich właściwości wytrzymałościowe. Stwierdzono również, że na wytrzymałość wytworzonych aglomeratów ma również wpływ skład ziarnowy granulowanego surowca.