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**MODELING THE PROCESS OF GRANULATION OF DUSTY-TYPE CLAY WITH
DIPPER METHOD ON A PELLETIZING GRANULATOR****I.A. Talybly¹, G.M. Samedzade¹, L.F. Masyeva², A.N. Mammadov^{1,2},
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Abstract: *In a disk - shaped two-section granulator with coaxial sides, the preparation of granules from clay powder by the action of drops from binder fluid of the same diameter was studied. Optimal granulation time in the first and second sections, the corner of inclination and the rotation speed of the granulator are established, which contribute to the production of rounded granules with a sphericity of at least 0.85 (85%) and the same particle size distribution. It was established that value of the granules formed is determined by the size of the supplied droplets of the binder fluid, and the strength increases with increasing diameter. However, with increasing granule size, the tensile strength decreases. It is shown that the initial stages of the granulation process have the greatest influence on the potential mechanical strength of granules. A mathematical model of the diameter dependence of the granule on the diameter of droplet is proposed to the binder solution. It was shown best coincidence of results by the proposed mathematical model and the experimentally obtained data.*

Keywords: *two-section granulator, powdery clay, droplet granulation, mathematical model.*

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Introduction

The granulation process of powdered materials by the pelletizing method is widely used in the production of fertilizers, metallurgy, construction materials, animal feed, food and medical preparations. Here with, various industrial equipment is used for granulation (drum, disk, dish-shaped, cupped, belt granulators, etc.) [1–4].

Regardless of the granulation method, in all cases, the formation and growth of granules occurs due to moistening of the contacting powder particles with a binder, which leads to agglomeration and coagulation of the particles. Obviously, the characteristics of the liquid phase have a significant effect on the process of granule formation and, in particular, on the diameter of the granules, due to which adhesive, capillary and surface forces appear which constrain the particles into lumps, nuclei, and primary granules. When applying a binder solution by spraying, the number of drops of solution falling on the same granule

or lump is difficult to determine, and therefore, it is difficult to establish correlations between the dimensional parameters of the final granules, as well as the dispersion of their dispersed composition and the parameters of the sprayed solution.

For obtaining granules of comparable diameter, especially for such a sticky material as clay, wiping machine were created, from where the material is squeezed out in the form of cut wet lumps, which are then granulated together with the powder in drum or disk granulators [5]. A method is proposed for granulating a powder moving using a horizontal belt conveyor on which drops of a binder solution fall, and the resulting primary lumps are then granulated in a drum granulator [6]. It was also known that the vibration granulator, in which drops are injected from a drop - sensor located at the bottom of a vibrating chamber [7]. A number of granulators have been described [8], in which

the authors strive to obtain spherical granules of a given composition and properties.

We developed a technology for producing granules from clay by the drip method on a plate granulator to obtain round pellets of the same size with a sphericity of 0.85 or more [9] considering the known

advantages and disadvantages of the above-mentioned granulator designs.

The aim of this work is to develop a mathematical model of the process based on the drip method of granulation of pulverized clay in the disk granulator to obtain granules with desired properties.

Research Methodology

The influence of the main process parameters on the size and strength of the formed granules was studied, namely, the angle of inclination of the disk to the horizon, the size of the granules from the size of the droplets of the supplied binder solution and the

time of rolling in an experimental study of the process of granulation of pulverized clay by a drop method. Researching were performed on a two-section disk granulator shown on the fig.1.

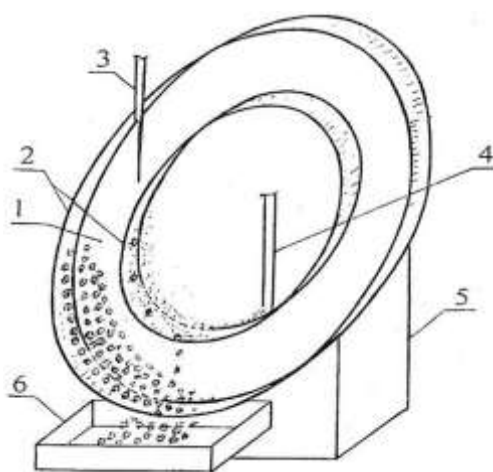


Fig. 1. Two-section disk granulator. 1 – plate surface, 2 – coaxial sides, 3 – capillary, 4 – drain, 5 – plate frame, 6 – hopper.

The diameter of the plate along the outer side 25 mm, high was 270 mm, and the diameter along the inner side 12 mm, high of the first section was 180 mm. Pulverized clay along the stock 4 was sent to the first section of the plate on the right, and drops of a given size from the capillary 3 were fed to a coalescing clay layer to the left of the center

(the plate rotates clockwise). The obtained granules after rolling with the participation of the powder are rolled into the second section and rolled for a predetermined time in the absence of powder. On fig. 2 was shown photographs (COSONY camera) of pellets rounded with powder (fig. 2 a) and further pelletized without powder (fig.2 b).



Fig.2. Pictures of granules: a - granules rounded with powder, c - granules rounded additionally without powder.

Raw granules were dried at 105–1100C and moisture was determined by weight loss. The moisture of individual granules was also

determined on a NETZSCH STA 449F3 derivatograph. Derivatives of granules and granular powdered clay are shown in fig. 3.

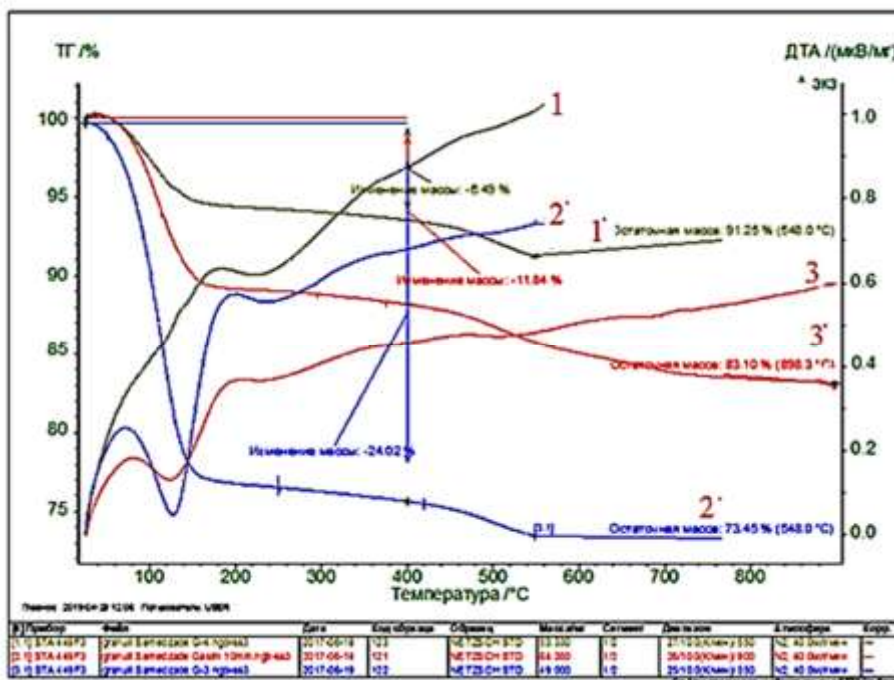


Fig. 3. Derivatives of granules and granular powdered clay.

1,1' – DTA and thermogram of granular powdery clay, 2,2' – DTA and thermogram of granules obtained by rolling for 20 sec. with powder, 3,3'-DTA and a thermogram of pellets pelletized for 10 seconds with powder and then 10 min. without powder.

As follows from the powder clay derivatogram, the moisture contained in the samples is released at 105–1750C and amounts to 5.7% (mass). With a further increase in temperature to 4500C, crystallization water begins to be released, which does not affect granulation. From the derivatogram of granules (fig. 3, curves 2.2 ') obtained by rolling for 20 seconds with powder, it follows that due to evaporation of moisture, weight loss is 22.97%. This means that, due to this excess moisture during pelletizing with clay powder, the granule can still be layered with powder and increase in diameter. Losing weight of a similar granule with an additional pelletizing of 10 min. without powder amounted to 10.9%, which corresponds to a minimum moisture, insufficient for further layering of clay particles and an increase in the volume of granules [9].

The strength of the granules was determined after natural drying at room

temperature and at 1050 ° C on a Fick appliance. The ordinary clay of the following composition (% wt.) was granulated: quartz - 45.0, muscovite 2M1 - 29.5, dickit 2M1 - 6.4, octadecasil -1.5, muscovite 2M2-17.6 determined on a “DU Phaser” X-ray diffractometer (BRUKER). X-ray data are shown in fig. 4.

The diameter of the drop supplied to the granulation was specified on the basis of their amount formed when one milliliter of water flows from the capillary. Since $4 / 3\pi r^3 \cdot k = 1000 \text{ mm}^3$, then

$$r = \sqrt[3]{\frac{3 \cdot 1000}{4\pi k}},$$

then the diameter of the drop $F_{\text{drop}} = 2r$ and $F_{\text{drop}} = 12.4091^3/1/k$ (1)

where F_{drop} – diameter of the drop, mm; k– the number of drops in one ml.

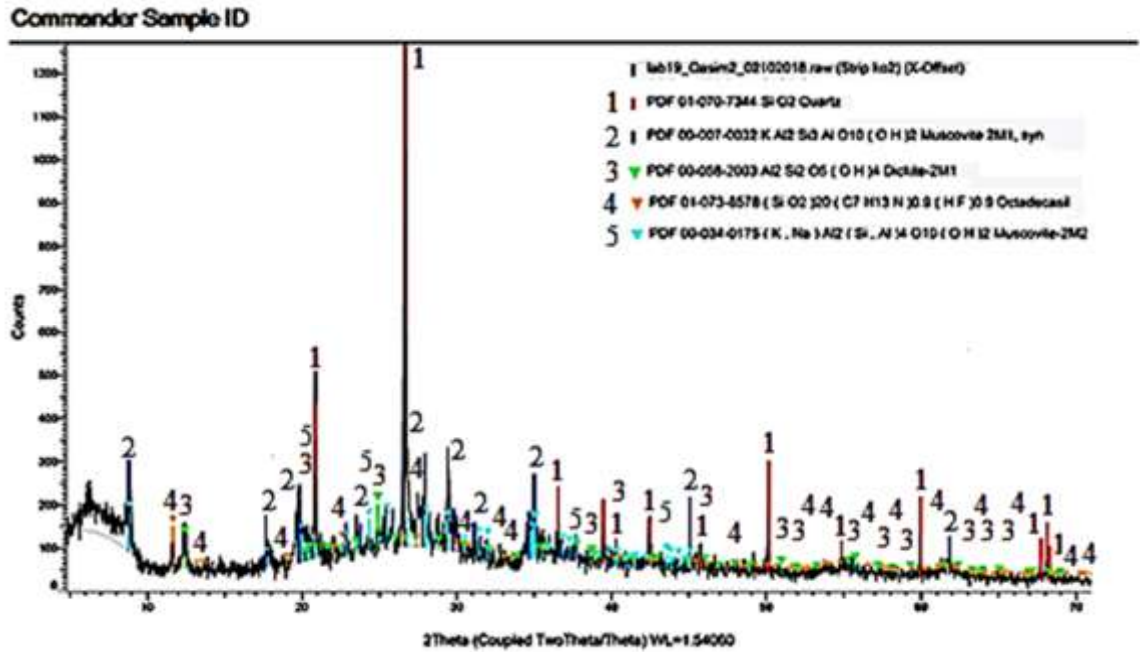


Fig. 4. Radiograph of granulated clay. 1 – quartz, 2 – muscovite 2M1, 3 – dickit 2M1, 4 – octadecasil, 5 – muscovite 2M2

Despite the predominantly stochastic nature of the mutual arrangement of particles arising during the granulation process, their collisions, adhesion adherence and the manifestation of capillary power, agglomeration and binding of powder particles into a single system (granule) with the participation of a binder are subject to certain patterns.

Thus, the formation and growth of granules from a powdery medium substantially depend both on the size of the dropping drops of the liquid binder and on its properties (viscosity, density, surface tension, etc.). A noticeable effect on the granulation process is also exerted by the mixing conditions in the apparatus, the fractional (dispersed) and chemical composition of the powder and its formality [13].

With intensive stirring at an early stage of granule formation, the change in mass of a single granule in time, by analogy with the law of acting masses, can be represented as

$$m \frac{dN}{dt} = N \bar{m} \quad (2)$$

where m is the mass of the granule; N is the number of particles; \bar{m} – mass of one particle.

Condition is always supported in a closed system.

$$m_i N > m_b N$$

If N_n the limit of the number of particles, bound by one drop, then $N_n \cdot m_b \geq m_i N$, that is, one drop can only bind N_n particles, reaching the limit size.

The thickness of the layering and the conditions for the completeness of the structure of the granule are determined by the moisture capacity or wettability of the surface. In this regard, in [10, 11], on the basis of the researching of the effect of droplet sizes on the formation of nuclei in granulators - mixers and the growth rate of granules, the concept of the time of liquid penetration into the powder layer, defined as

$$\tau_p = 1.35 \frac{v_0^{1/3}}{\varepsilon^2 \rho} \cdot \frac{\eta_s}{\sigma_{\text{ac}} \cos \theta_0} \quad (3)$$

Where v_0 - the volume of a drop of liquid; ρ - pore radius; ε - porosity; η_s - fluid viscosity; θ_0 - wetting corner; σ_{ac} - liquid surface tension.

For observing the interaction of the drop with particles of dusty clay, particles of clay powder were inflicted to the hydrophobic flat surface of solid paraffin, and then a drop of water with a diameter $F_{\text{drop}} = 2.86 \text{ mm}$.

Introduction of powder particles into the drop and their migration in the direction of its movement were observed when rolling a drop of water through the powder particles.

As a rule, clay particles are deployed in the droplet volume when droplets move.

If particles are applied to the surface of a droplet, then their glide along its surface to the base is observed. At the same time, clay particles, layering on top of each other and remaining on the outer sphere of the droplet, gradually get wet and penetrate into the formed sphere. This effect is due to the interaction of two oppositely directed forces - the surface tension of water droplets and adsorption capillary power. Penetrating to the drop, a particle literally settles to the bottom (on the surface of particles in a drop). It is obvious that the force arising from the compression of a drop of clay particles prevails, and is sufficient to introduce particles into the drop.

Thus, in falling drops into a porous medium, particles of dusty clay sliding along the shell, the drop captures, moistens, and draws small particles into the sphere without losing its integrity. This process ends with enveloping the entire surface of the droplet with clay particles and the droplet converted into a granule, continues to slide to the bottom of the granulator plate over the surface of the clay particle layer. Time which measured from the drop falling onto the surface of a rolling layer of clay particles and its rolling out from the layer onto the surface in the form of a granule does not exceed one second.

Obviously, the excess of water molecules in the drop rushes to its surface and here, under the influence of gravitational and centrifugal forces, further layering of new clay particles occurs on the surface. Considering migration of unbound water molecules to the

granule surface, it is necessary to take into account the significant effect of capillary pressure (RC) on the fluid flow. Distribution of particles in the granule determines the volume of the porous body where the excess liquid is located. The fluid in the capillary is affected by the resulting pressure due to capillary forces (R_d) and power friction F_{tr} . Equation of change in the momentum of fluid motion in the capillary has the form:

$$\frac{d(v_{жс} \pi \rho^2 z_f \theta)}{dt} = -\rho_k \pi \rho^2 - F_{тр} \quad (4)$$

Where z_f - meniscus coordinate, \bar{v} - the average fluid velocity coinciding with the velocity of the meniscus, r_k - capillary radius, v_j density, ρ - pore radius [12]:

$$\bar{v} = dz_f / dt \quad (5)$$

We must note that, the time required for impregnation for length of the capillary l , is proportional l^2 and inversely proportional to the radius of the capillary ρ :

$$t = (l) \frac{2\eta}{\sigma_{жс} \cos \theta \cdot \rho} \cdot l^2 \quad (6)$$

Resulting force of gravity and the centrifugal force resulting from the rotation of the granulator plate contributes to the direction of the excess moisture down a vertical trajectories. During the rotation of the rolling granule, this trajectory of movement is distributed along the entire perimeter of the granule.

Resulting of collisions of the granules between each other and with the plate wall, further compaction of the granules and squeezing of excess moisture onto the surface. When granule is almost dry in rolling, without adding new portions of powder, moistening of their surface is observed after 40 - 70 seconds.

Influence during of the rolling with and without powder on moisture and granule size

Influence of rolling granules with powder in the first section of the granulator on the value of their diameter and moisture was determined for droplets of the same diameter $F_{drop} = 2.72$ mm. After supplying the droplet, the granules were rolled for 10–60 sec. In a sample of ten obtained granules, the diameter

and humidity were determined. Data are shown in Table 1.

As, can be seen from the data in Table 1. with an increase during of rolling from 10 sec. up to 60 sec. the diameter of the granules increases slightly from 4.03 to 4.26 mm, while the wet decreases to 17.57%.

Table 1. Dependence of the diameter and moisture of the granules during their pelletizing with powder at a droplet diameter of 2.72 mm.

Time roll. sec	Quant. granules	Weight raw granules q	Weight dry granules q	Wet		Middle diameter granules mm	Sphericity granules	Weight 1 gr.
				В Г	b% В%			
10	10	0.559	0.440	0.119	21.29	4.03	0.876	0.044
20	10	0.618	0.495	0.123	19.90	4.01	0.848	0.049
40	10	0.623	0.513	0.110	17.66	4.10	0.823	0.051
60	10	0.683	0.563	0.120	17.57	4.26	0.875	0.056

In [1, 3] it was proposed to display the dependence of the growth rate of granules on the initial moisture content in the form of the following equation:

$$x_t = x_0 e^{m(\omega - \omega_0)} \quad (7)$$

Where x_0 – size of the original granules; relevant ω_0 , mm; x_t – the size of the final granules, mm; ω_0 – initial moisture content; m– sensitivity of granular materials to content of moisture change (m, x_0 и ω_0 – determine experimentally and characterized the granularity of the material) [1].

With the drip method of granulation, the diameter of the granules directly dependent on the water content in forming granules, from the diameter of the droplet and from the time of droplet-granule formation. This dependence is well described by the proposed formula:

$$F_{gr} = F_{drop} \cdot e^{m+ln t} + k \cdot 10^{-3} \text{ at } t < 1 \text{ min.} \quad (8)$$

Where F_{gr} – diameter of granule, mm; F_{drop} diameter of drop, mm; m and n– determined by calculation and consistent with experimental data; t– granulation time, min.; κ – the quality of drops in 1 ml. solution; $ln t^n$ – characterizes the change in the size of the granules depending during of rolling. Proceeding on the experimental data for calculating the diameter of the granules accepted, $m=0.338$, $n=0.026$.

Fig. 5 were presented the values of the dependence of the diameter of the granules from the time of rolling the droplet — granules with and without powder, curve 1 is calculated, curve 2 is experimental. As can be seen from the picture, the calculated data coincide with the experimental data and are reflected by the one curve.

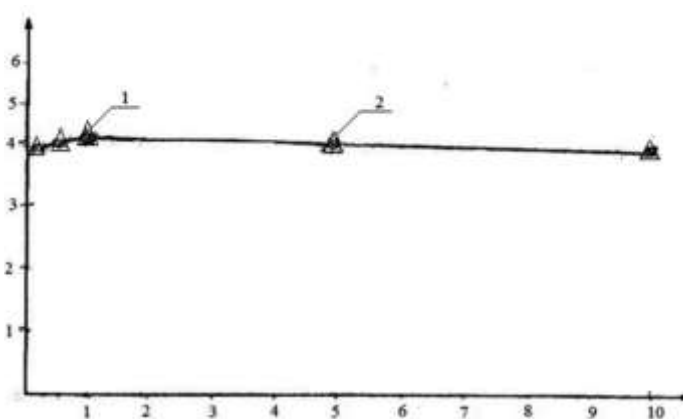


Fig. 5. Curves of dependence diameter of the granules from the time of their rolling:
1-Δ – experimental, 2 - ● – calculated

All researching shown that during the interval of 10-60 seconds the size of the granules when rolling in a powder layer increases with 3.82 to 4.12 mm i.e. by 7%

(look table 2.)

Obtained drop-granules look like balls with a crumbly surface from the adhering clay powder, picture 3a, which crumbles when

shaking. Obviously, for hardening the surface granules without the participation of clay of the granules, it is necessary rolling the powder, which was noted earlier in [9].

Table 2. Dependence of the granules diameter on the diameter of the drops and the time of their pelletizing obtained by calculation and practically.

Rolling time		k – 19drop/ml $F_{drop} = 4.65\text{mm}$		k – 45drop/ml $F_{drop} = 4.89\text{ mm}$		k – 82drop/ml $F_{drop} = 2.856\text{ mm}$		k – 95drop/ml $F_{drop} = 2.72\text{ mm}$	
Sec.	Min.	Fgr Calc. mm	Prac. mm	Fgr Calc. mm	Prac. mm	Fgr. Calc. mm	Prac. mm	Fgr. Calc. mm	Prac. mm
10	0.1667	–	–	–	–	–	–	3.895	3.82
20	0.3333	–	–	–	–	–	–	3.96	4.0
40	0.6667	–	–	–	–	–	–	4.03	4.08
60	1	6.61	6.81	5.05	5.18	4.24	4.26	4.07	4.12
	5	6.34	6.39	4.85	4.89	4.07	4.06	3.915	3.94
	10	6.23	6.06	4.76	4.73	4.00	4.0	3.85	3.86
	15	6.17	5.95	4.72	4.61	3.967	–	3.56	3.81

Additional rolling granule without powder was carried out in the outer, second section of the granulator for 5–15 min. Results of measuring the diameter of the granules from the duration of rolling without powder are given in table 2.

It was found that experimentally determined data are well described by the proposed formula:

$$F_{gr} = F_{drop} \cdot e^{m+nlnt} + k \cdot k^{-3} \quad (9)$$

where $nlnt$ - characterizes the change in the size of the granules depending on the time of

rolling; m and n – constant depending on the physicochemical properties of the material; F_{drop} – diameter a drop of water, mm; k – number of drops in 1 ml of water. Calculation was performed at $m = 0.338$ and $n = 0.026$.

Investigation Strength of Granule

Finding the optimal pelletizing regime, we studied the influence of the pelletizing time for 3,5,10 and 15 minutes on the shape and strength of the obtained droplet granules. The data obtained are shown in table 3.

Table 3. Influence of the rolling time on the tensile strength granule with granule strength at various diameters.

Time granulation min.	Diameter of the granules, mm					
	3-3.35		4-4.5		5-5.5	
	Granule strength					
	n/granule	MPa n/mm ²	n/granule	MPa n/mm ²	n/granule	MPa n/mm ²
3	7.4	0.223	8.09	0.143	9.163	0.106
5	7.5	0.226	8.53	0.150	8.991	0.104
10	7.69	0.232	8.87	0.156	9.653	0.112
15	7.84	0.236	9.02	0.159	9.996	

As we see, the main strength of the droplet-granule during rolling is formed in the first 2–3 min. after its formation. The best results in the form of droplet -granules and

their strength were obtained by rolling in the first section with powder for one minute, and then in the second section without powder, for 5–10 min. Following results of given table,

with increasing size granules, the strength of the granules increases, but the ultimate strength decreases.

Discussion of the results

All research showed that the mechanism of layering powdered clay on the surface of the granule, from the upper position on a disk granulator to the lower, doesn't change [1, 9–11], and is realized due to the water from the granule onto its surface during compaction. The densest packing of the granule is obtained by rolling the granules in powder for one minute, followed by rolling them in the second section without powder for 5–10 minutes. As so, size of the drops supplied to the granulation is the same, accordingly, the value of the obtained granules doesn't change. Thus, with the drop method, the surface of the granules is moistened from the inside of the granule and the growth of the granule is ensured by the stock of this internal moisture. During granulation in powder for a minute and subsequent granulation without powder, the surface of the obtained granules is not wetted and, consequently, the cost of drying and subsequent firing is reduced. During granulation in powder for a minute and subsequent granulation without powder, the surface of the obtained granules is not wetted and, consequently, the cost of drying and subsequent firing is reduced.

As the formation of granules ends in 1

second and granule rolls on powder surface, it is precisely at the appropriate speed that droplets should be supplied to the granulation. An increase in the frequency of the droplet feed leads to the formation of granules with a dumbbell shape. Best granulation results were obtained with drop method obtained at a plate slop of 57–580 and a rotation speed of 27 rpm. At a drop feed rate of 1.4–1.6 drops / sec. it is possible to obtain up to 95% round-shaped granules with a sphericity of at least 0.85.

The configuration of the surface of granules after the first section requires additional rolling, which is carried out in the second section of granulator and leads to the formation of granules with a smooth round surface. Value of the granules is determined by the size of the supplied droplets of the binder fluid and the strength increases with increasing diameter. However, with increasing granule size, the tensile strength decreases.

The proposed mathematical model for determining the size of granules depending on the diameter of the droplet describes well the results of pelletizing in excess powder by formula (8) and when pelletizing without powder by formula (9).

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BOŞQABVARI QRANULYATORLARDA DAMCI ÜSULU İLƏ GİL TOZLARININ DƏNƏVƏRLƏŞDİRİLMƏSİ PROSESİNİN MODELƏŞDİRİLMƏSİ

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İki bölməli qranulyatorada gil tozundan bağlayıcı maye damlacıqlarının təsiri eyni ölçülü qranulların alınması prosesində öyrənilmişdir. Ən azı 0.85(85%) sferikliyi olan yuvarlaq qranulların alınması üçün qranulyatorun birinci və ikinci bölmələrinin optimal qranulyasiya müddəti, meyl bucağı və qranulyatorun fırlanma sürəti müəyyən edilmişdir. Yaradılmış qranulların ölçüsü bağlayıcı mayenin damlalarının ölçüsü və yaranma sürətindən asılıdır. Bununla birlikdə, müəyyən ölçüyə çatdıqda qranulların davamlılığı azalır. Qranulyasiya prosesinin ilkin mərhələlərinin qranulların potensial mexaniki gücünə ən böyük təsiri olduğu göstərilmişdir. Bir qranulun diametrinin bir damcı möhkəmləndirici məhlulun diametrindən asılılığının riyazi modeli təklif olunur. Təqdim olunan riyazi model ilə təcrübi nəticələr arasında yaxşı uyğunlaşma müşahidə olunur.

Açar sözlər: iki bölməli qranulyator, gil tozları, möhkəmləndirici maye damcıları, riyazi model.

МОДЕЛИРОВАНИЕ ПРОЦЕССА ГРАНУЛИРОВАНИЯ ПЫЛЕВИДНОЙ ГЛИНЫ КАПЕЛЬНЫМ МЕТОДОМ НА ТАРЕЛЬЧАТОМ ГРАНУЛЯТОРЕ

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В тарельчатом двухсекционном грануляторе с коаксиальными бортиками исследовано получение гранул из порошка глины воздействием капель связующей жидкости одинакового диаметра. Установлены оптимальное время грануляции в первой и второй секциях, угол наклона и скорость вращения гранулятора, способствующие получению округлых гранул со сферичностью не менее 0.85 (85%) и одинаковым гранулометрическим составом. Установлено, что величина формирующихся гранул определяется размером подаваемых капель связующей жидкости, а прочность возрастает с увеличением их диаметра. Однако с увеличением размера гранул предел прочности уменьшается. Показано, что на потенциальную механическую прочность гранул в наибольшей степени оказывают влияние начальные стадии процесса грануляции. Предложена математическая модель зависимости диаметра гранулы от диаметра капли связующего раствора. Показано хорошее совпадение результатов, рассчитанных по предложенной математической модели и экспериментально полученных данных.

Ключевые слова: *двухсекционный гранулятор, порошкообразная глина, капельная грануляция, математическая модель.*