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THE EFFECT OF WETTING ON SILICA FLOUR GRANULATION

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The effect of changes in surface tension and degree of liquid jet break-up as well as final moisture content of the bed on changes in particle size distribution during wet drum granulation was described in the paper. The tumbling bed of loose material (silica flour) was wetted at a constant volumetric flow rate, using a system of two pneumatic spray nozzles. Different values of surface tension of the binding liquid (distilled water) were obtained due to the application of a surfactant Rokanol L4P5. In every trial samples of the feed were taken from the drum at specified time intervals and on this basis particle size composition was determined.

Key words: drum granulation, surface tension, granulation kinetics

INTRODUCTION

Granulation is one of the methods of processing powder materials into granulated products which are more suitable for storage, transport and further processing. The process consists in the formation and growth of particles in a mobile bed of material. When the wetted material tumbles in the drum, interactions occur between solid particles and liquid droplets depending on the properties of particular media. In the case of granulation, important parameters are both particle size composition of the tested material, physicochemical properties of the liquid wetting the bed, the method of its dosing and mutual quantitative relations (moisture content).

Iverson et al. (2001) assumed that properties of the tested product depended on three stages of granulation (Fig. 1). The authors defined dimensionless numbers that determined the course of two first stages and developed a so-called map of granulation regimes useful in the assessment of the process mechanism. However, such maps are not suitable to predict properties of the granules, in particular their size distribution.

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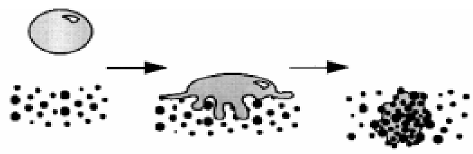
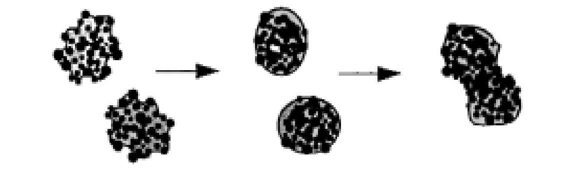
Wetting and nucleation



Growth and consolidation



Disintegration and grinding of granules

According to Newitt and Conway-Jones (1958), granules can appear in four states, depending on the amount of liquid present in the intraparticle spaces:

a) pendular – single liquid bridges between particles,
b) funicular – with free space in the granule filled partly with air,
c) capillary – with free space between granules filled entirely with liquid, however with dry outer surface,
d) drop-like – with particles enclosed totally in liquid.

All these states can occur during a single granulation process. During wetting of the material a drop falling onto the bed causes local overwetting and forms a drop-like state with material particles. As a result of bed tumbling and attaching new not wetted particles to the formed nucleus, the newly formed granule is transformed into a porous, loosely packed agglutination of material particles in the pendular state. Next, as a result of collisions of particles against each other and drum walls in the tumbling bed, the air is gradually removed from the granule which makes that it is transferred into funicular and next capillary state. As a result of a further condensation of particles in the granule, the liquid is pressed out from the granule which causes formation of big unstable agglomerates and determines the end of the process.

While analysing silica flour, Gluba et al. (2004) found that particle size distribution of the material subjected to granulation had a significant effect on the process. They observed that the bigger is the grain diameter, the smaller is the granulation rate and that mean granule diameter increased with an increase of the mean droplet diameter.

When searching for a binding agent, Ennis et al. (2000) analysed the forces that occurred during collisions of two spherical particles. They proposed a viscosity Stokes number whose value is inversely proportional to binding liquid viscosity and does not depend on its surface tension. On the other hand, Nienow (2005) modified this theory and proved that surface tension of the liquid phase should be introduced into it. Basing on experiments, he declared that the surface tension had a bigger influence on the granulation process than viscosity itself.

Due to complexity of problems related to the effect of wetting parameters on the granulation process, further studies are necessary on the kinetics of granulation of raw materials with different physical properties in various wetting conditions and then some generalisation should be searched for.

THE AIM OF RESEARCH

The aim of research was to describe the effect of changes in surface tension and degree of wetting liquid jet break-up as well as final moisture content of the bed on tumbling agglomeration kinetics in a horizontal drum granulator.

CHARACTERISTICS OF TESTED MATERIALS

The tested material was a commercially available fraction of silica flour MK 0.075, produced in Strzeblow Mineral Mine at Sobotka. The particle size composition of this material was determined using a laser particle size analyser ANALYSETTE 22. The mean flour particle size d_z= 0.024 mm was determined on the basis its size distribution.

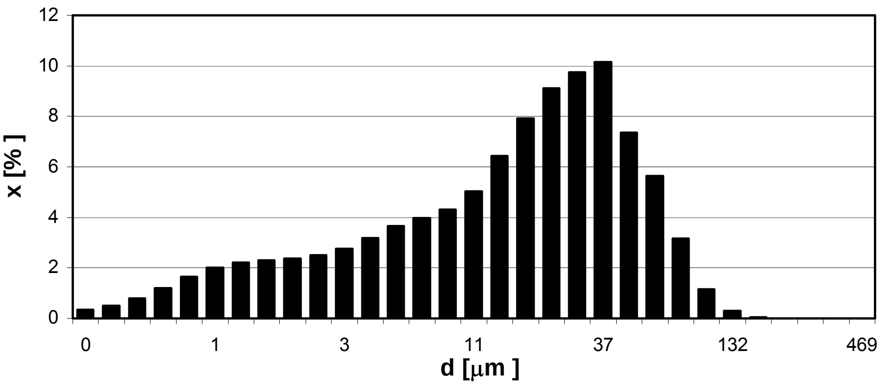


Fig. 1. Particle size distribution of silica flour MK 0.075

Wetting liquids used in the experiments were distilled water and two water solutions of Rokanol L4P5 at different concentrations. Rokanol L4P5 is a trade name of polyoxyalkyl-glycol ether of saturated lauryl alcohol, produced by PCC ROKITA S.A. in Brzeg Dolny. This compound was used to decrease the surface tension of distilled water (Table 1). A small concentration of Rokanol in the solution and its properties similar to water (e.g. density ρ = 0.99 g/cm^2), cause that other features of distilled water do not change significantly.

Table 1. Liquid surface tension

	Tested liquid		
	Distilled water	Water solution of Rokanol	
		0.01%	0.03%
Surface tension $\sigma \cdot 10^{-3}$ [N/m]	71.97	54.79	37.61

THE SCOPE OF INVESTIGATIONS AND MEASURING METHODS

In the whole experimental cycle the rate of wetting liquid flow through nozzles was constant and equal to Q_w=12·10^{-3} m^3/h. Changes in the liquid jet break-up (drop size) were caused by changes in the rate of air flow through the nozzles in the range Q_p=2.5 to 4 m^3/h which provided four different coefficients of jet break-up q defined as the ratio of liquid flow rate Q_w to air flow rate Q_p (Table 2). The drop size distribution in the broken-up jet at specified parameters of nozzle operation, was measured by a laser drop size analyser DANTEC. Investigations were made for defined final moisture content of the bed w = 0.19, 0.195, 0.2 and 0.205 (kg water/kg dry material).

Table 2. Parameters of spray nozzle operation

Q_w	Q_p	q
[m^3/h]	[m^3/h]	[-]
0.012	2.5	0.0048
0.012	3	0.004
0.012	3.5	0.0034
0.012	4	0.003

The process of granulation was carried out batch-wise in a horizontal drum with longitudinal baffles (1), of diameter D = 0.5 m and length L = 0.4 m. In the whole experimental cycle the rotational speed of the granulator was constant and equal to n = 0.25 s^{-1}. The mass degree of drum filling with raw material k = 0.1 was also constant and determined in reference to bulk density of loosely packed material. The drum was driven by an electric motor (3) through a cogbelt and coupling. For adjustment and control of the rotational speed, an inverter (4) and revolution meter were used, respectively. The granular bed in the drum was wetted by two pneumatic nozzles, Spraying System Deutschland GmbH (2). They were mounted on a separate stand (5) and introduced axially to the apparatus through a hole in the cover. The flow rate of liquid supplied from the tank (7) placed at the height 3.5 m on the drum axis, was set by a liquid flow controller COLE-PARMER LC-500 (6a), while the flow rate of air supplied by an air compressor (8) was set by a mass air flow controller AALBORG GFc47 (6b).

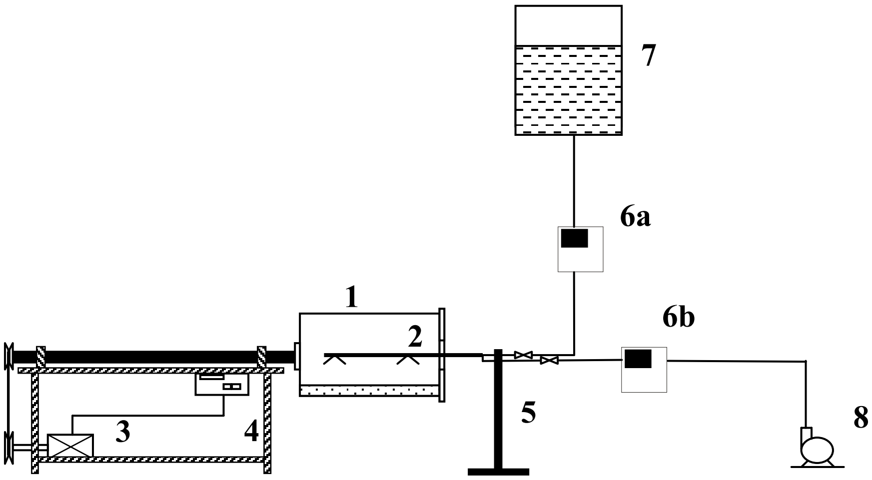


Fig. 2. Diagram of the measuring station. 1- drum, 2- spray nozzles, 3- motor, 4- inverter, 5- stand, 6a- liquid flow controller, 6b- air flow controller, 7- water tank, 8- air compressor

At the initial stage of experiments, the flour was dried in order to remove moisture from it. After supplying a proper amount of flour to the drum, the first stage of the process, i.e. wetting, started. After dosing the whole wetting liquid on the tumbling bed at assumed operation parameters of the spray nozzles, the second stage proceeded, i.e. granulation. In time intervals (t = 0, 4, 8, 16, 24 and 32 [min]) constant for each trial, representative samples were taken from the drum by means of a specially constructed device. The samples were subjected to a particle size analysis. The first sample was taken immediately after finishing the wetting process, and the last one at the moment when the process was completed (t = 32 min). The samples were dried at the temperature 338 K for 24 hours, and next they were weighed, which enabled the analysis of granulation kinetics.

RESULTS

Based on the analysis of the samples taken immediately after finishing the wetting process, it was found that the bed contained both not granulated raw material and a specified fraction of nuclei and weak granules. The amount and size of particles obtained at this stage of the process depend mainly on the size of liquid drops and final moisture content of the bed (U_s – total share of particular granule fractions; D_{mean} – mean granule diameter).

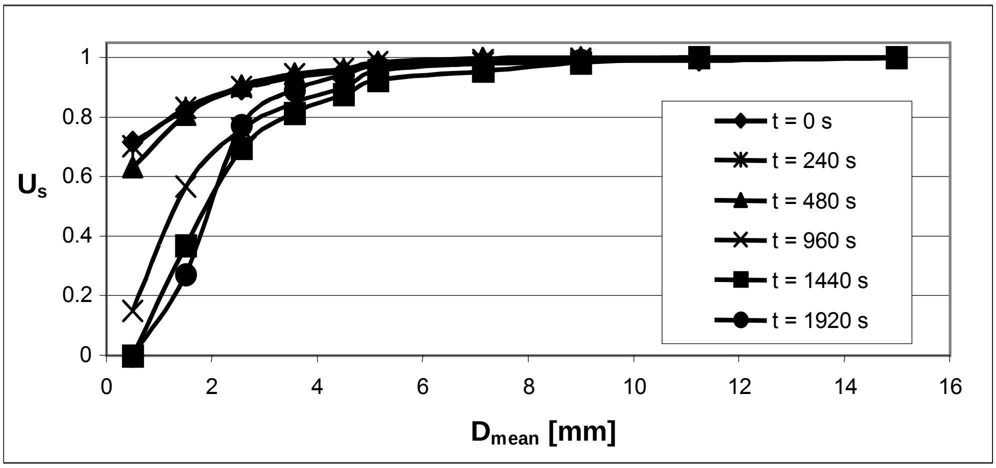


Fig. 3. Change in the particle size composition of the feed during granulation for q = 0.002; w = 0.19kg/kg; σ = 54.79·10^{-3} N/m

When analysing the change of particle size composition (Fig. 3) one can find that the process of granulated material formation is not uniform in time. During the wetting process and at the initial stage of granulation the increase of mean granule size is due mainly to the nucleation and aggregation of not granulated feed mass on earlier formed nuclei. In this period the rate of changes in the average granule size increases. At subsequent stages of the granulation the smallest fraction is reduced until its absolute exhaustion. In this period of the granulation process a maximum rate of the granule size increment is observed. Then, due to collisions the granules gradually condense which causes that water is pressed out of the granules to their surface. When water appears on the surface, a further growth of granule size is observed. A dominating mechanism of granule growth at this stage is consolidation and coalescence. This is a period in which the rate of changes of mean granule size decreases.

It was found that the rate of changes taking place in the granulated bed depended on wetting conditions. Figure 4 shows a change in the mean granule size during the process for different degrees of wetting liquid jet break-up. It follows from this Figure that with an increase of the jet break-up the rate of agglomerate growth decreases. This process can be affected by both the size of nuclei formed during the nucleation and capillary forces in liquid bridges that connected particles in the granules. It was also observed that the effect of liquid jet break-up on the granule size diminished with a decrease of liquid surface tension.

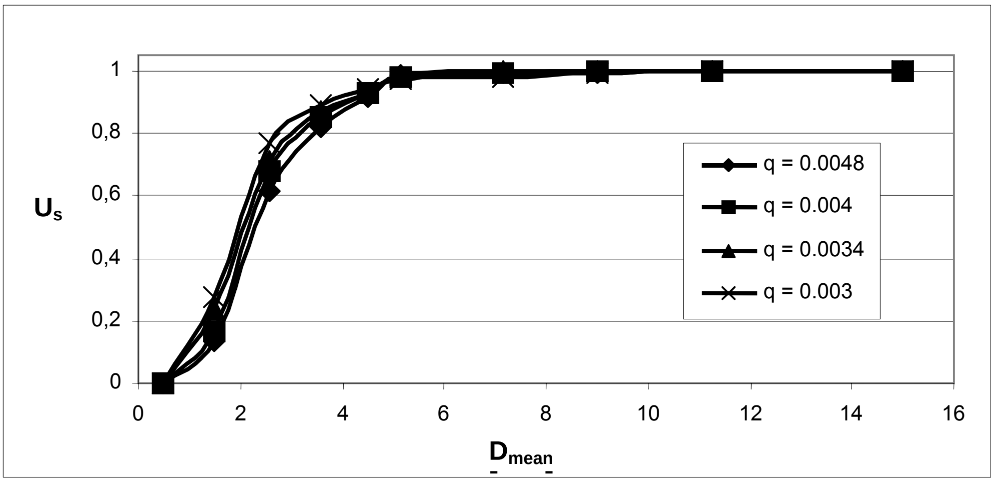


Fig. 4. The effect of liquid jet break-up on the change of particle size composition of the tested material at t = 1440 s; w = 0.2 kg/kg; $\sigma = 54.79 \cdot 10^{-3}$ N/m

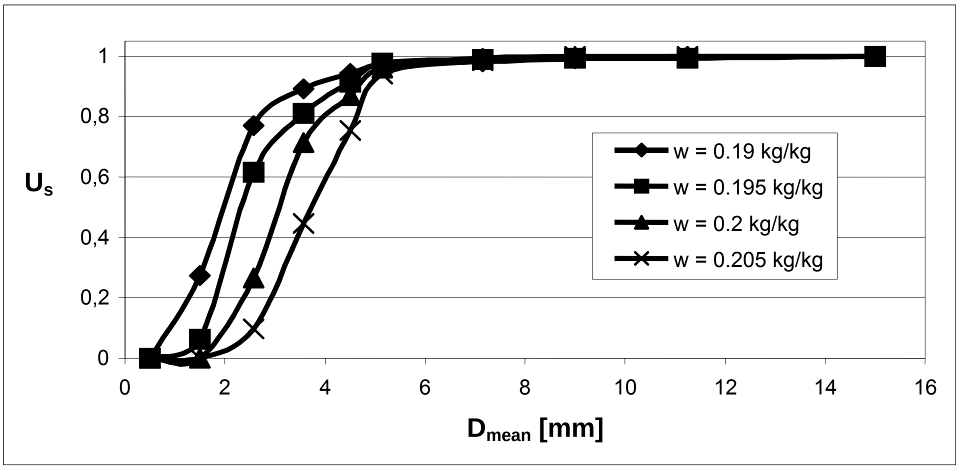


Fig. 5. The effect of final moisture content of the bed on changes in the particle size composition of the tested material at t = 960 s; q = 0.0032; $\sigma = 37.61 \cdot 10^{-3}$ N/m

The higher final moisture content of the granulated bed causes a significant increase of mean particle diameter of the granulated product (Fig. 5). This result is determined by two mechanisms: at the initial stage of granulation a bigger amount of water in the bed causes higher elasticity of the nuclei and makes that their collisions result in the formation of big agglomerates, further during the granulation, excess moisture which is pressed much faster from the granules causes that material which is still not granulated quickly agglomerates.

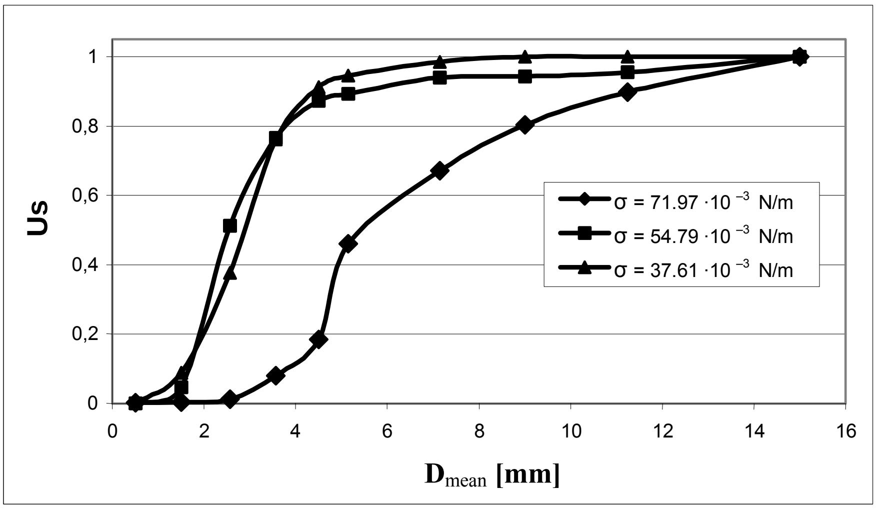


Fig. 6. The effect of changes in surface tension on particle size composition of the feed at $q = 0.0048$; $w = 0.2$ kg/kg; $t = 1920$ s

As shown in Figure 6, the decrease of wetting liquid surface tension has a significant effect on the size of formed granules. It was observed that an increase of Rokanol concentration in the wetting liquid solution results in a remarkable decrease of the agglomerate growth rate. This relation follows most probably from a lower resistance of the agglomerates and domination of the mechanisms of granule breaking and attrition over growth and consolidation. Of special importance is the fact that an increase of Rokanol concentration in the solution, and consequently a decrease of its surface tension, does not cause such a big change in the particle size composition of the product. Probably, for smaller granules the decomposition processes are balanced with the processes of their growth and consolidation.

CONCLUSIONS

1. Results of experiments show a significant effect of bed wetting parameters on the kinetics of wet drum granulation.
2. With an increase of mean liquid drop diameter the mean granule diameter in the bed decreases. The effect of liquid jet break-up on granule size decreases with a decrease of the liquid surface tension.
3. A decrease of the liquid surface tension causes a decrease of the rate of agglomerate growth and unification of the particle size composition in the bed.
4. With an increase of feed moisture content an increase of the process rate and granule size was observed.

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Heim A., Gluba T., Obraniak A., Gawot-Młynarczyk E., Błaszczyk M., *Wpływ nawilżania na granulację mączki kwarcowej,* Physicochemical Problems of Mineral Processing, 40 307-315, (2006) (w jęz. ang.).

Celem pracy było zbadanie wpływu zmian napięcia powierzchniowego cieczy nawilżającej, wielkości kropel oraz wilgotności końcowej złoża na właściwości granulowanego złoża otrzymanego w procesie mokrej granulacji bębnowej. Materiałem badawczym była mączka kwarcowa pochodząca ze Strzeblowskiej Kopalni Surowców Mineralnych w Sobótce. Wykorzystano frakcję oznaczoną symbolem MK 0,075 dla której skład ziarnowy frakcji określono za pomocą laserowego analizatora wielkości ziaren „ANALYSETTE 22". Proces granulacji prowadzono w sposób okresowy w poziomym bębnie o średnicy D = 0,6 m i długości L = 0,4 m obracającym się ze stałą prędkością obrotową n = 15 obr/min. W przeprowadzonych doświadczeniach jako ciecz nawilżającą zastosowano wodę destylowaną z dodatkiem Rokanolu L4P5. W całym procesie nawilżania natężenie dopływającej cieczy, ustalane za pomocą regulatora przepływu cieczy COLE-PARMER LC-500, było stałe i wynosiło $Q_w = 12\cdot10^{-3}$ m^3/h. W celu uzyskania różnych wielkości kropel nawilżających stosowano zmienne natężenia przepływu powietrza przez dysze, ustalane za pomocą masowego regulatora przepływu powietrza AALBORG GFc47, w zakresie $Q_p = 2{,}5 \div 4$ m^3/h. Badania prowadzono przy ustalonych wartościach wilgotności w = 0,19; 0,195; 0,20; 0,205 [kg/kg]. W stałych dla każdej próby momentach czasowych pobierano z bębna reprezentatywne próbki które poddawane były analizie sitowej, a następnie suszeniu i ważeniu co umożliwiło wykonanie analizy kinetyki granulacji. Uzyskane wyniki wykazały istotny wpływ warunków nawilżania na właściwości granulatu otrzymanego w wyniku granulacji bębnowej.