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THE EFFECT OF WETTING PARAMETERS ON MECHANICAL STRENGTH OF GRANULATED MATERIAL

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Results of the investigation of the effect of changes in surface tension, wetting liquid drop size and final moisture content of the bed on the strength of granulated material are presented in the paper. The tested material was silica flour. After each run, representative feed samples were taken from the drum and on their basis the strength of the granulated product was determined by means of abrasion and compression tests. The abrasion tests were conducted in a horizontal drum of diameter D'=0.2 m and length L'=0.14 m, with perforated walls, where the bed was destroyed during tumbling. Trials were made for steady rotational speed of the drum n'=26 r.p.m. and for constant times of testing t=30 to 180s. The compression tests were conducted between two pressing plates up to the moment when a single granule was destroyed between the pressing surfaces.

Key words: drum granulation, granulated product strength

INTRODUCTION

Mechanical strength of granulated products is one of the most important features that determine their further processing possibilities. Formation of an agglomerate with specific properties requires a proper granulation method and application of relevant process parameters with special reference to wetting conditions.

The bed of material saturated with liquid, which is a complicated three-phase system, requires that many parameters having an influence on material strength should be taken into account.

Narrow interparticle spaces in the bed of comminuted material are capillary in nature, hence the liquid present in the bed forms characteristic liquid bridges with curved surfaces that are a result of liquid surface tension (Fig. 1).

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Fig. 1. Schematic of a liquid bridge between two solid surfaces

Conditions of wetting the granulated bed affect the character of capillary interactions in the layer of loose material, which depends on the amount of water in the point of particle contact, type of contact and the number of contacting points in the unit of material volume. Hence, the type of forces that bind particles in the granule depends on liquid content in the interparticle space. Newitt and Conway-Jones (1958) distinguished four basic states of distribution of the liquid present in a granule (Fig. 2):

- pendular free spaces between particles are only partly filled with liquid, single liquid bridges appear between solid particles. Strength of this agglomerate is induced by the presence of discrete liquid bridges in the points of contact or due to closeness of particles;
- funicular the number of liquid bridges increases, free space in the granule is partly filled with liquid;
- capillary all pores between particles are filled with liquid, concave meniscus appears on the outer surface. The granule strength depends on capillary subatmospheric pressure; between particles there are no other substances – particles can interact;
- drop-like –material particles are suspended in the liquid. The strength of granules depends only on liquid surface tension.



Fig. 2. States of the granule in the presence of the liquid: suspended (pendular), linear (funicular), capillary, drop-like

In literature there are many publications dedicated to studies on granule breakage. From the point of view of granulated product quality, studies on granulated product strength are significant for the processes of transport, storage and applications of these materials.

238

Pepin et al. (2001) studied the strength of wet granules in relation to the content and type of a binding liquid. Suzuki et al. (2001) analysed the process of microcrystalline cellulose (MCC) granulation and observed that hardness of the granulated product increased with an increase of water added.

Gluba (2001) tested the compression strength of granulated dolomite flour and found that with an increase of drop size of liquid supplied by the pneumatic nozzles, the strength of the granulated product was increasing. He also observed that the granulated material obtained during drop-wise wetting was characterised by lower strength than in the case of wetting with the biggest drops from the nozzle. Walker et al. (2003) tested the crushing strength of a NPK fertiliser. Based on analyses, they observed that the final product containing a bigger amount of water, prior to drying, was characterised by a higher strength. Bika et al. (2005) studied the crushing strength of granules formed from lactose and mannitol that were wetted with different liquids. They found that addition of a surfactant to a binding liquid caused a significant decrease of strength of the dried granulated product.

AIM OF THE STUDY

The study was aimed at the assessment of the effect of changes in surface tension, wetting liquid drop size and final moisture content of the bed on the strength of granulated product obtained in the process of tumbling agglomeration.

EXPERIMENTAL SET-UP AND METHODS

The granulation process was carried out batch-wise in a horizontal drum granulator of diameter D = 0.6 m and length L = 0.4 m. The drum was driven by an electric motor via a toothed gear and belt transmission. In the whole experimental cycle constant rotational speed of the granulator n = 20 rpm and constant volumetric filling of the drum with raw material k = 0.1 (16.125 kg), in relation to bulk density of loosely packed material, were used.

A tested material was silica flour (MK 0.075) produced in Strzeblow Mineral Mine at Sobótka. The mean flour particle size was d_z = 0.024 mm. Wetting liquid was distilled water with addition of a surfactant (Rokanol L4P5 – polyoxyalkyl-glycol ether of saturated lauryl alcohol). Changes in water surface tension γ , caused by Rokanol added to the wetting liquid are shown in Table 1.

	Rokanol L4P5 concentration in the binding liquid [%]		
	0	0.01	0.04
γ·10 ⁻³ [N/m]	71.97	54.79	35.04

Table 1. Surface tension of the wetting liquid

Fine-grained material in the drum was wetted, while tumbling, by means of two pneumatic spray nozzles introduced axially to the drum. Experiments were made at a constant rate of wetting liquid flow through the nozzles $Q_w=12\cdot10^{-3}$ m³/h and at determined values of air flow rates in the range $Q_p = 2.5$ to 4 m³/h, which enabled different degrees of liquid jet break-up, determined by the coefficient $q = Q_w/Q_p$, and consequently, different values of mean drop size d_k (Table 2). Experiments were carried out at three final moisture contents of the bed in the range w = 0.19 to 0.20 [kg water/kg dry material].

$Q_{\rm p} [{\rm m}^3/{\rm h}]$	q [-]	$d_{\rm k}$ [mm]
2.5	0.0048	0.154
3.0	0.0040	0.143
3.5	0.0034	0.134
4.0	0.0030	0.128

Table 2. Spray nozzle operation parameters

Having completed the granulation (32 min), a representative product sample of mass ca. 1 kg was taken from the drum, dried, separated into size fractions on sieves to determine the particle size composition, and then on this basis the strength of granulated product was determined.

In order to test the resistance to attrition, a granulated material sample of mass ca. 0.5 kg was placed in a horizontal drum of diameter D' = 0.2 m and length L' = 0.14 m equipped with perforated walls (Fig. 3), where during bed tumbling the sample was destroyed. Tests were carried out at determined rotational speed of the drum n' = 26 rpm. For each sample the experimental time t = 30 to 180s was constant and after every experiment the mass of abraded material was determined (d_g < 1.5mm).



Fig. 3. Set-up for testing attrition resistance of the granulated material: 1- perforated drum, 2- balance, 3-motor, 4- inverter

240

To investigate compression strength, 10 granules in the shape close to spherical were taken from each fraction, and next each granule was placed separately between parallel compressing plates (mobile and immobile). The test lasted until the moment when the granule was destroyed between the compressing plates (Fig. 4).



Fig. 4. Experimental set-up for testing compression strength of the granulated product: 1- compressing plates, 2- lever, 3- motor, 4- spring, 5- winding reel, 6- potentiometer, 7- torquemeter.

RESULTS

Abrasion resistance of the granulated product was estimated using the grindability index that represents the loss of material by measuring an increment of the mass of abraded material (particles of diameter $d_g < 1.5$ mm).

The grindability index was determined from the relation:

$$W_{S(t)} = \frac{m_w - m_{S(t)}}{m_w} \cdot 100\%$$
(1)

where: $m_{S(t)}$ – mass of abraded material, after time *t*,

 $m_{\rm w}$ – total mass of the sample charged to the drum.

Compression strength of the granulated product was estimated on the basis of the force of granule breakage during compression. The force was calculated by measuring the torque moment on the winding reel shaft, recorded by the torquemeter in the moment of sample destruction. The following relation was used:

$$P = \frac{M_s}{D_{bn}/2} \tag{2}$$

where: D_{bn} – winding reel diameter [m],

 $M_{\rm s}$ – moment recorded in the time of granule destruction [Nm].

The above calculation of the breaking force with reference to the granule size, allowed us to determine the conventional compressive stress described by the formula:

$$\sigma_s = \frac{4P}{\pi d_g^2} \tag{3}$$

where: $d_{\rm g}$ – granule diameter [m].

Results of the experiments showed a significant influence of the binding liquid drop size (liquid jet break-up) on the abrasion process. Examples of the relations presenting changes in the grindability index W_s during abrasion of granulated product are given in Fig. 5. It follows from this diagram that when the bed is wetted with big drops the strength of granulated product is higher. This can be explained by the mechanisms of granulated product formation. In the case of wetting the bed with big drops, bigger nuclei can be formed which are then transformed into granules. Wetting with small drops leads to the formation of granules by coalescence which causes that sphericity of the obtained agglomerates is lower and air is enclosed inside the formed granules, which much reduces their mechanical strength.



Fig. 5. Change of $W_{\rm S}$ during attrition for different wetting liquid drop sizes (w = 0.19; $\gamma = 71.97 \cdot 10^{-3}$ N/m)

Figure 6 shows changes in the breakage force for different diameters of granules obtained in tumbling agglomeration. It follows from the figure that the compression strength of the granulated product increases linearly with a mean granule size. Results of experiments show also that even the use of small amounts of a surfactant added to the wetting liquid (0.01%) causes a decrease of compression strength of the granules. Most probably this is caused by formation of weaker liquid bridges (lower surface tension) than in the case of granulated material wetted with distilled water. It was observed also that for bigger granules a further increase of Rokanol concentration in the binding liquid caused only a slight decrease of the tested material strength.



Fig. 6. Comparison of curves $P = f(d_g)$ for different values of surface tension of the binding liquid (w = 0.195, $d_k = 0.143$ mm)

To determine the effect of final moisture content of the bed on the strength of tested granulated material, curves $\sigma_s = f(d_g)$, presented in Fig. 7, were compared. When analysing the graph below, we can observe that with an increase of granule size the breaking stress induced by sample compression decreases. It was also found that with an increase of the bed final moisture content the strength of tested material increased. Lower strength of the granulated product formed from less humid bed can be caused by higher non-homogeneity of binding particle structures inside the granules. A reason may be the presence of air bubbles, beside liquid bridges (pendular state), which weaken the granulated product significantly. In the case of granules formed from the bed of higher final moisture content, the air is forced from the granules by the binding

liquid faster (funicular or capillary state), which causes enhancement of interparticle bonds, and consequently, an increase of the tested sample strength. Results of the tests showed also that an increase of the final moisture content of the granulated bed best improved the strength of granules with small diameters, while in the case of bigger granules differences in the breaking stresses that appeared during compression were minimal.



Fig. 7. Comparison of curves $\sigma_s = f(d_g)$ for different final moisture contents of the feed (d_k=0.143 mm; γ =54.79·10⁻³ N/m)

CONCLUSIONS

- 1. Mechanical strength of a product formed during wet drum granulation depends significantly on wetting parameters. Very important is final moisture content of the granulated bed, wetting liquid drop size (jet break-up) and surface tension.
- 2. An increase of drop size of the liquid supplied by pneumatic nozzles leads to the formation of a product with higher mechanical strength.
- 3. A decrease of liquid surface tension causes a decrease of granule strength.
- 4. An increase of the final moisture content of the bed contributes to the formation of granules with higher mechanical strength.

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W pracy przedstawiono wyniki badań wpływu zmian napięcia powierzchniowego, wielkości kropel cieczy nawilżającej oraz wilgotności końcowej złoża na wytrzymałość otrzymanego granulatu. Jako materiał badawczy zastosowano mączkę kwarcową pochodzącą ze Strzeblowskiej Kopalni Surowców Mineralnych w Sobótce (MK 0.075). Granulację prowadzono w sposób okresowy w poziomym bębnie o średnicy 0,6m i długości 0,4m, przy stałej prędkości obrotowej n=15 obr/min. Złoże materiału sypkiego nawilżano w czasie jego ruchu przesypowego, przy stałym objętościowym natężeniu przepływu cieczy $Q_{\rm w} = 12 \cdot 10^{-3} \text{ m}^3/\text{h}$ (woda destylowana z dodatkiem Rokanolu L4P5), za pomocą zestawu dwóch pneumatycznych dysz rozpyłowych. W celu uzyskania różnej wielkości kropel (stopnia rozbicia strugi) stosowano zmienne natężenie przepływu powietrza przez dysze $Q_p = 2.5 \div 4 \text{ m}^3/\text{h}$. Badania prowadzono przy ustalonych wartościach wilgotności końcowej złoża w=0.19÷0.2 [wag]. Po każdej próbie pobierano z bebna reprezentatywne próbki wsadu, na podstawie, których określano wytrzymałość otrzymanego granulatu za pomocą testów na ścieranie oraz ściskanie. Badanie wytrzymałości na ścieranie, prowadzono w poziomym bębnie o średnicy $D^{2}=0.2$ m i długości $L^{2}=0.14$ m zaopatrzonym w perforowane ścianki, gdzie w czasie ruchu przesypowego złoża następowało jego niszczenie. Próby prowadzono dla ustalonej prędkości obrotowej bębna n'=26 obr/min. oraz dla stałych czasów prowadzenia testu $t=30\div180s$. Badanie wytrzymałości na ściskanie prowadzono między dwoma płytkami ściskającymi, próba trwała do momentu zniszczenia pojedynczej granulki miedzy ściskającymi ją powierzchniami. Uzyskane wyniki pokazały istotny wpływ zmian warunków nawilżania złoża, podczas aglomeracji bębnowej, na wytrzymałość mechaniczną otrzymanego granulatu.