

BALLING AND GRANULATION

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1. A hydraulic setting cement is incorporated as the binder additive. The green pellets are partially hardened in silos, where they remain for about 24–36 hr, and the hardening process is completed during storage in stockpiles.
2. The binders are silica, lime, slag, or cement. The balls are somewhat dried, if necessary, and then cured in steam autoclaves. During the hydrothermal treatment lime and silica react to form hydrosilicate gels, which act as binders.
3. Hardening is effected by carbonation instead of by steam autoclaving.

VII. Granulation of Fertilizers

A typical fertilizer granulation loop is shown in Fig. 26. The feed comprises dry raw materials, raw materials in form of slurry or solution, and recycled solids. The granules from the granulator pass through a drier, where the water is removed and the capillary bonding mechanism is replaced by crystal bridges, forming strong agglomerates. The dry product is screened into onsize granule grade, which leaves the circuit. The undersize, and a fraction of the onsize if necessary, is fed back to the granulator. The oversize is also recycled after passing through a crusher. At steady state the rate of exit of the product grade granules from the circuit is equal to the rate of formation of new agglomerate entities. Irrespective of the kind of process employed, there are in general two kinds of sources of new granule formation (H3). One of these is internal to the granulator: nucleation of discrete

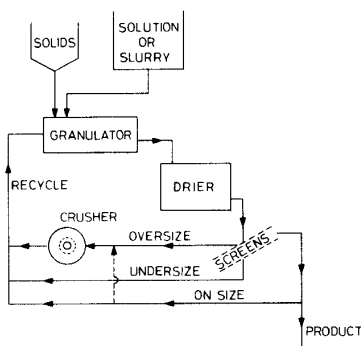


FIG. 26. Fertilizer granulation loop.

species from the particulate feed and by attrition and breakage of the tumbling charge. The other kind is external to the granulator and includes breakdown of granules in the drier and crushing in the crusher. The crusher may turn out to be the primary source of new seed species in a circuit operating in the snowballing mode.

The solution of the fertilizer salts in water, frequently referred to as the *solution phase*, acts as the bonding liquid. The solid feed to the granulator is relatively coarse: the average size often exceeds 1 mm. The amount of the liquid in the charge usually lies in the range 20–30% volume, which is definitely less than the critical saturation value. A major objective in fertilizer granulation is to maximize the product grade material in a narrow size-distribution range, typically 1–4 mm or even 1.5–3 mm (S9).

Depending on the process conditions, the fertilizer granules are formed by either random coalescence or snowballing mechanisms or both. In general, one of these growth modes is found to predominate. However, the coalescence mechanism, also known simply as agglomeration, is more common. Some fertilizer formulations that are difficult to agglomerate by coalescence may be amenable to granulation by the snowballing mechanism (also referred to as onion skinning or layering).

A. GRANULATION IN COALESCENCE MODE

1. *Solution-Phase Theory*

From studies based on model batch systems by Sherrington (S9) (coarse sands bonded with saturated solutions of fertilizer salts), Butensky and Hyman (B9) (glass beads bonded with an aqueous solution of chemical grout), as well as by Newitt and Conway-Jones (N2) and Capes and Danckwerts (C5) (moist coarse sands), one may draw the following broad conclusions concerning granulation of coarse fertilizer feed in the presence of insufficient bonding liquid. In the initial stage coalescence is the primary mechanism of granule growth. In the later stage, when the specific surface area of the granules falls below a critical value, the agglomerates become “surface dry.” The granules are now rigid or brittle, do not deform on collision, and do not possess sufficient surface plasticity to form a strong bond between two colliding pellets. The final product invariably contains some apparently ungranulated material in the same size range as the feed powder. Presumably, this material arises from abrasion of loosely held particles from the dry surface of the large granules, and perhaps by the destruction of the weak pellets. The abraded powder and broken fragments are

continuously picked up by snowballing and layering. The granules eventually attain a steady-state size distribution, which is effectively invariant of agglomeration time, charge loading, and the technique employed for mixing liquid with the feed solids. The mean size of the viable granules in dynamic equilibrium is a function of the amount of liquid, provided the granule-to-particle size ratio is not too large. The mean size increases and the proportion of the ungranulated material decreases with an increase in the solution phase content.

Sherrington (S9) and Butensky and Hyman (B9) have presented a model that relates the equilibrium granule size to the amount of the granulating liquid present. Consider a spherical agglomerate comprising a number of single size spherical particles, whose idealized cross section is shown in Fig. 27. It is assumed that the granule surface consists of an outer spherical shell of thickness g times the particle radius, where g is an unknown factor whose value is about unity. A fraction S of the void space is filled with liquid, the remainder being entrapped air. The weight of the bonding liquid is

$$(\pi/6)S\epsilon\rho_L(D - gD_p)^3 \quad (96)$$

where ρ_L is the liquid density. The weight of the solids per granule is

$$(\pi/6)(1 - \epsilon)\rho_S D^3 \quad (97)$$

Therefore, the weight of liquid per unit weight of solids is

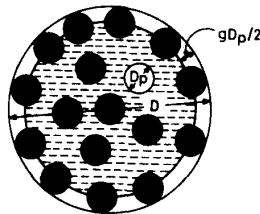
$$W = W_\infty[1 - g(D_p/D)]^3 \quad (98)$$

where

$$W_\infty = S\epsilon\rho_L/(1 - \epsilon)\rho_S \quad (99)$$

is the limit of W when the relative diameter or the size enlargement ratio D/D_p approaches infinity. In other words, W_∞/S is the weight of liquid per unit weight of solids required to fill the voids in an infinitely large granule. Equation (98) by Butensky and Hyman (B9) reduces to the expression given by Sherrington (S9) when D/D_p is large and $S = 1$. Obviously, the model is

FIG. 27. Idealized cross section of a granule that is deficient in liquid. [From Butensky and Hyman (B9).]



valid only when the size enlargement ratio exceeds a minimum value of 3 or 4 (B9).

Actual measurements of the liquid content have shown that the larger granules have more liquid associated with them than the average and the smaller granules have less, in agreement with Eq. (98). Assuming that S and g are independent of pellet size, the liquid content may be related to the pellet size distribution in the following manner (B9):

$$W = W_{\infty} \sum_i [1 - g(D_p/D_i)]^3 w_i \quad (100)$$

where w_i is the weight fraction of solids in granules of size D_i . Next, we define an appropriate mean granule size D^* so that

$$W = W_{\infty} [1 - g(D_p/D^*)]^3 \quad (101)$$

Combining Eqs. (100) and (101) and solving for the mean relative size D^*/D_p , we get

$$D^*/D_p = \frac{g}{1 - \left\{ \sum_i [1 - g(D_p/D_i)]^3 w_i \right\}^{1/3}} \quad (102)$$

A comparison of the measured mean relative size with the model in Eq. (101) is shown in Fig. 28. The material was monosize glass beads. The data fit the model quite well, with the exception of fine 0.038-mm powder. It is evident that the steady-state size distribution is a function of the liquid content, and consequently, as shown by Sherrington (S9), there is an optimal granulating liquid for maximum granulation efficiency, that is, percentage of the product-grade material.

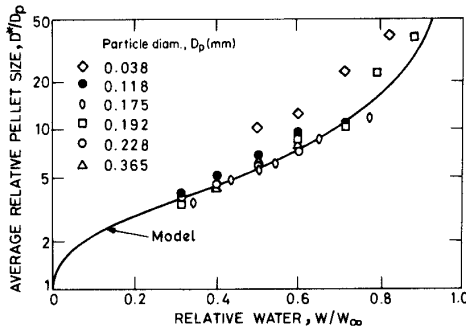


FIG. 28. Effect of the relative liquid content on the average relative pellet size. [From Butensky and Hyman (B9).]

2. *Granulation Loop*

The presence of the recycle loop in fertilizer granulation process makes it prone to surging and drifting, which coupled with long lag times give rise to difficulties in maintaining a stable operation of the plant. A rational strategy for countering the surging and drifting effects can be established from the solution phase theory (S9). For example, if there is a momentary increase in the liquid feed to the granulator, this will cause an excess production of the oversize granules for a short period (Eq. 101). This, in turn, may result in a higher average feed size when this impulse of excess oversize recycles and completes the loop. As a consequence, a follow-up surge, leading again to increased production of oversize, will travel through the loop even though the liquid feed has settled down to its normal level. It may take several hours before this kind of surging smoothens out. Again, if for some reason the crusher product becomes even slightly coarser, a steadily accelerating drift in the process will occur, leading to increasing production of the oversize granules. The drifting can be quite serious unless corrective measures, including turn-down of the liquid feed to the granulator, are taken promptly.

Control of the liquid phase to the granulator is the primary control variable available to the plant operator. The minimum amount of water that enters the feed along with the new raw material is determined first by the fertilizer formulation. It may be less or more than that required to form sufficient liquid phase for optimal granulation efficiency. In the former case an adjustable extra water supply is all that is needed; the recycle is now determined by the granulation efficiency attained in the system and the plant is “granulation limited” (S9). In the latter case it may be necessary to recycle some of the onsize granules, preferably after crushing, in order to reduce the average feed size to the granulator. The plant is now “water balance limited” and much more difficult to control (S9).

B. GRANULATION IN SNOWBALLING MODE

1. *Model Studies*

In circuits operating in the snowballing mode, there is an increased tendency for large-size feed particles and large recycled aggregates of particles to act as seeds rather than constituents of the deposited layer surrounding another granule. Moreover, individual particles and their clusters of size greater than about 0.7–1.2 mm always act as seeds rather than agglomerate with each other (S10).

Sherrington (S10) has carried out interesting simulation studies on laboratory scale using 1.2–1.5-mm glass beads as the seeds and finer sand as the layering material. In the absence of glass beads the fraction of oversize material in the granule size distribution (> 3.5 mm) increases steadily with increasing liquid content. When the beads are present and liquid content is low, the oversize production is quite small and a large amount of sand remains ungranulated. Initially, the principal effect of adding increasing amounts of the liquid phase is to increase the fraction of on-size granules at the expense of ungranulated sand. When the liquid content required to layer all the sand is exceeded, growth occurs by coalescence with a rapid increase in the formation of oversize granules. It is estimated that 30% seeds in the charge is about the lower limit below which snowballing cannot be achieved. As shown in Fig. 29, addition of seeds to the sand increases the maximum proportion of onsize agglomerates at the optimal liquid content which ranges from about 40 to 80% volume. The maximum yield of the onsize granules is not very sensitive to the proportion of seeds present, but the liquid content for the maximum yield decreases with an increase in the number of seeds. These results suggest that the realization of coalescence or snowballing as the principal growth mechanism, as well as the dynamic behavior of the system in the latter case, depends in a complex manner on the interactions between recycle ratio, size of recycled material, and liquid-to-solid feed ratio.

The liquid-phase requirement for the snowballing mechanism may be computed in the following manner (S10). It is assumed that the liquid withdrawal is negligible (i.e., $q = 0$) and the fractional saturation in the deposited

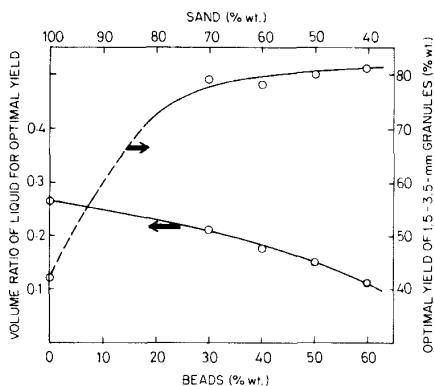


FIG. 29. Optimal liquid content and optimal yield of on-size granules for different fractions of seeds in the charge. [From Sherrington (S10).]

layer is unity. Let subscript 1 pertain to the snowballed granule or the deposited layer portion of the granule. The weight of the layered solids in a granule of size D_1 is

$$(\pi/6)(D_1^3 - D^3)\rho_{s,1}(1 - \epsilon_1) \quad (103)$$

and the weight of the liquid in the granule is

$$(\pi/6)(D_1^3 - D^3)\rho_L\epsilon_1 + (\pi/6)D^3\rho_L\epsilon S \quad (104)$$

where S , the fraction of pores in the seed filled by liquid, is the degree of ingress of liquid into the seed. Let L be the weight ratio of seed to layered solids:

$$L = D^3\rho_s(1 - \epsilon)/(D_1^3 - D^3)\rho_{s,1}(1 - \epsilon_1) \quad (105)$$

From these relationships the weight of the liquid per unit weight of solids in a granule is

$$W = \rho_L(\rho_s J_1 + L\rho_{s,1}JS)/(\rho_{s,1}\rho_s + L\rho_{s,1}\rho_s) \quad (106)$$

where

$$J = \epsilon/(1 - \epsilon) \quad (107)$$

and

$$J_1 = \epsilon_1/(1 - \epsilon_1) \quad (108)$$

The snowballed granule diameter is

$$D_1 = D[1 + \rho_s(1 - \epsilon)/L\rho_{s,1}(1 - \epsilon_1)]^{1/3} \quad (109)$$

When $\rho_{s,1} = \rho_s$, which is generally valid, Eq. (106) becomes

$$W = (\rho_L/\rho_s)(J_1 + LSJ)/(1 + L) \quad (110)$$

and when $S = 0$

$$W = (\rho_L/\rho_{s,1})[J_1/(1 + L)] \quad (111)$$

Equations (106) and (109) give expressions for the liquid content and granule size in a snowballing granulation system.

2. Granulation Loop

Sherrington (S10) has presented a static model of the granulation loop operating in the snowballing mode. The model combines the solution phase theory in Eq. (106) with the material balance across the granulator. The recycle ratio defined as recycle/raw solid feed, is given by

$$U = (2\rho_s/SJ_1\rho_L)(1 + X) + J_1 YX/SJ - J_1/SJ \quad (112)$$

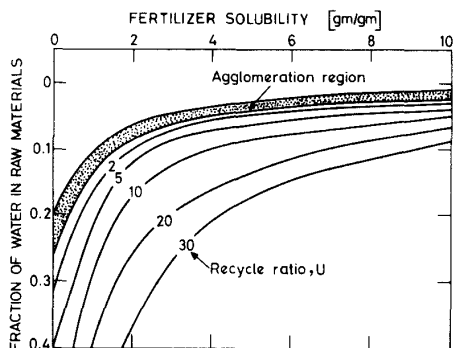


FIG. 30. Simulated behavior of fertilizer loop operating in the snowballing mode. [From Sherrington (S10).]

where ρ_s is now density of the fertilizer, X the weight ratio of fertilizer to water in the fertilizer solution at the granulation temperature, and Y the weight ratio of water to fertilizer in the incoming raw materials. Results of numerical simulation using typical values for the various parameters are presented in Fig. 30 in terms of the variation of the recycle ratio with fertilizer solubility and fraction of water with the raw materials. In the snowballing region the recycle ratio is always larger than in the coalescence region, and it increases with increasing solubility and increasing water fraction. The recycle contours are bunched together when the fertilizer solubility is high. The hatched area pertains to the coalescence region in which snowballing is not possible, although agglomeration can occur outside this region.

Han (H3) and Han and Wilenitz (H4) have also presented steady-state models of fertilizer granulators based on population balance on the granules in the process loop operating in the snowballing mode. From the viewpoint of process control some interesting interrelationships between various recycle ratios, crusher speed, crusher product size, and the granule growth rate have been established.

VIII. Miscellaneous Topics

In this section three miscellaneous topics in the area of agglomeration shall be discussed, namely, dry pelletization, spherical agglomeration in liquid suspension, and spontaneous or inadvertent agglomeration of fine particles.