History of Granulation and State of the Art Granulation Techniques

Curtis Griffin

\textsuperscript{1}Pegasus TSI, 5310 Cypress Center Drive Tampa Fl, 33609 USA
History of Fertilizer

In the early 1800’s, it was discovered that phosphorus promoted growth in plants and animals. The first agricultural fertilizer came from bones which contained the element phosphorus. The success of that early fertilizer required a more commercial approach. Some of the first patents for the manufacture of “superphosphate” were issued in 1842. By 1845, the early bone crushing factories were converted into “superphosphate” factories and the commercial phosphate fertilizer industry was launched.

Phosphate rock was first mined in England in 1847 for use as a fertilizer. In 1881, Captain J. Francis LeBaron, of the Army Corps of Engineers, discovered Florida’s treasure in phosphate pebbles in the Peace River.

Mono-Ammonium Phosphate (MAP) production began in 1920. Production of granular “superphosphates” by the Oberphos process began around 1929.

In the 1950s, fertilizer manufacturing facilities were relatively small and produced fertilizers tailored to the soil needs of area farmers, commonly within a 100-mile radius.

Basic laboratory and pilot plant work on the production of Di-Ammonium Phosphate (DAP) was done by the Tennessee Valley Authority (TVA) between 1950 and 1954. In 1956 Frank Nielsson of TVA patented the Ammoniator-Granulator. The TVA work resulted in larger scale granulation production with many of the concepts still used today. (See Figure 1)

Figure 1: Frank Nielsson Patent of the Ammoniator-Granulator
Early Technology and Granulation Processes

Eyman Process
This process uses a batch mixer with modified flights and a special sparger for the ammoniation of the solution. Either superphosphate or potash is used in granular form in the desired size range which is then combined with ammoniated solutions containing 6% water. Sulfuric acid is used as needed to provide the heat of reaction for drying. The formulation supplies just enough liquid to cement the fines in a thin layer on the original granules. The granular product from the batch mixer is cooled in a rotary cooler and then goes to storage.

Glaspey Process
This process is regarded as typical of ones in which the batch mixer is used for ammoniation only and granulation takes place in a dryer operated at high temperatures. The formulations are designed to avoid the generation of sticky mixtures that could cause operational issues. Less sulfuric acid is used than in other processes as the ammoniating solutions have lower water content. With this type of formulation, granulation must take place in the dryer as the low temperature in the batch mixer prevents granulation. A higher temperature in the dryer is used to fuse soluble salts and produce enough liquid phase for granulation.

Davison Trenton Process (Pug Mill)
The Davison Trenton process is the best known method that uses a pug mill for ammoniation. Most of the pug mills used today, are of the twin shaft type. Solid raw materials and recycled fines are fed into the inlet end of the pug mill, and liquids are injected under the bed. The kneading action of the pug mill gives a harder, stronger granule. Other advantages are that high ammoniation rates can be achieved and more variation in operating conditions can be tolerated. Among the disadvantages are high maintenance costs, high power requirements, poor fume control, and problems with non-uniform distribution of liquids under the bed. Granulation starts in the pug mill and is controlled by formulation, recycling fines, or adding water. In some cases, the pug mill discharges to a separate granulator; in most cases, however, it discharges to the dryer where additional granulation occurs. The dryer is usually of the co-current type. From the dryer, the product goes to a cooler and then to screens. The oversize is crushed and returned to the screen and the undersize is recycled to the pug mill. (See Figure 2)

![Figure: 2 Pug Mill](image-url)
**Swift Process**
The Swift process was developed and patented by Swift and Company; it involves the use of the Swift reactor for ammoniation and granulation. The reactor is a continuous rotary drum; the first section of the drum has a lifting flight zone. The lifting flights are staggered and designed to shower the dry material through the gas stream. Ammonia and the ammoniated solution are injected through a nozzle mounted in the seal plate of the feed end. Sulfuric or phosphoric acid is injected through an opening around the ammonia nozzle. The reaction of the acid generates steam which serves as the carrier gas. The granular product from the process will go directly to a cooler or to a dryer and then a cooler. The downstream equipment such as screening, crushing and recycle follow the typical design.

**Pan Granulators**
Pan granulation consists of a disc fitted to a peripheral wall which is rotated while in the horizontal position, additional feed and moisture is added to the pan, the moisture is typically in the form of fine sprays. Scrapers are used to prevent buildup of material on the disc and control the flow pattern of material on to the disc. Granulation occurs on the disc and when the granules attain the desired size, they are discharged from the pan. The damp granules are fed to a dryer and then to a screen. The major parameters controlling the pan output and movement of material on the pan are the pan diameter, the angle of incline from the horizontal, speed of rotation, and height of the peripheral wall. These parameters need to be optimized to assure correct granulation. (See Figure 3)

![Figure 3: Pan Granulator](image)

**Dorr-Oliver Process**
In the original Dorr-Oliver process, the ammoniation was carried out entirely in reaction vessels. Three reactors were used in a series operating at 0.6, 1.4 and 1.85 mole ratio respectively. The slurry from the final reactor flowed by launder to one or more pug mills or blungers. Because of the relatively insoluble nature of the slurry at 1.85 mole ratio, large amounts of water (30% or higher) were necessary to enable the slurry to flow. As a consequence, these early plants operated with a large recycle ratio of up to 12 to 1. Some refinements were made to the process whereby partial ammoniation was carried out in two vessels and the remaining ammonia added in the pug mill. This technique enabled the recycle ratio to be reduced to 8 to 1. (See Figure 4)
TVA Basic Process (Drum)
A major break-through came in 1956 when Frank Nielsson of TVA patented the Ammoniator-Granulator. This invention allowed large quantities of ammonia to be injected beneath the rolling bed of wet solids in a rotary drum with reasonable efficiency. In the TVA process only one “preneutralizer” or “reactor” vessel was used, operating at the point of maximum solubility (mole ratio 1.4-1.45) to minimize the slurry moisture (16-20%). This enabled the recycle ratio to be reduced to about 5 to 1. Modern plants have demonstrated that, with some tweaking of process conditions, this basic process can be operated at a recycle ratio of about 4 to 1. The basic unit consists of an open, slightly inclined rotary cylinder with retaining rings at both ends and with a scraper mounted inside the shell. A rolling bed of solid material is maintained in the unit, and liquids are introduced through horizontal, multiple outlet distributor pipes set lengthwise of the drum under the bed. Most of the drums used at that time were in size range between 5 and 15 ft. in length and 5 and 8 ft. in diameter. (See Figure 5)
Granulation Mechanism

Layering
In this process, ammonium phosphate slurry is sprayed onto the surface of the recycle or "seed" particles to form an additional layer. Each time a seed particle is recycled, an additional layer is added and the granule size increases. This mechanism is typical of high recycle processes where a granule makes many passes through the granulator before being removed as product. Product formed by layering has good hardness and is spherical. (See Figure 6)

![Figure 6: Layering](image)

Agglomeration
In this process, recycled particles are cemented together by the fertilizer solution forming salt bridges between individual particles. This mechanism is typical of low recycle processes. Product formed by agglomeration is much less spherical in appearance, more difficult to dry since the moisture is deep within the granule, and more prone to breakage as the bonds are not strong. (See Figure 7)

![Figure 7: Agglomeration](image)
Product Quality Standards

The industry quality standard is typically 2 by 4 mm granules between 93-95% with SGN 225 – 300 and UI 50 – 60.

**Size Guide Number (SGN)**
The median granule diameter multiplied by one hundred. The size at which 50% of the product is retained expressed in millimeters multiplied by one hundred.

\[ \text{SGN} = d_{50} \times 100 \]

**Uniformity Index**
The ratio of the small granules (particles retained at 95%) to large granules (particles retained at 10%) multiplied by one hundred.

\[ \text{UI} = \frac{[95\% \text{ retained}]}{[10\% \text{ retained}]} \times 100 \]

A UI of 100 means that all the granules are the same size.

(See Figure 8)

Figure 8: DAP
Present Day State of the Art Granulation Equipment and Operational Factors Affecting Granulation

**Reactor/Pre-Neutralizer**
The granulation process starts in the reactor, good control of the reactor is critical to controlling the downstream parameters that impact granulation. Granular MAP/DAP are produced by a slurry process that begins by adding a ratio of 54% and 30% phosphoric acid to the reactor. The 54% and 30% mixture is added at the correct ratio to give a total of 40% into the reactor. Ammonia is then added at a mole ratio of 1.42 for DAP and .6 for MAP forward titration to produce a slurry. The slurry is then pumped to the granulator to complete the reaction and start the main granulation process. The design of the reactor is critical for good slurry production and to minimize citrate insoluble (CI) losses. CI losses increase with residence time and acid impurities such as iron, aluminum and magnesium. To minimize CI loss, the state of the art reactor design has a smaller diameter in the lower section to reduce retention time.

**Granulator**
The main design parameters for good granulation in the granulator are, ammonia sparger location, slurry header location, slurry spray nozzle configuration, granulator speed, down leg support locations and shell cleaning mechanism (typically rubber panels).

**Typical design Criteria:**
- Retention time 1.5 – 2.0 minutes
- Length to Diameter Ratio 2.3
- Bed volume, % Drum Volume 12-20
- Slope 3/8 – 5/8
- Peripheral speed ft/min 285
- Discharge dam height 25% of drum diameter
- Recycle Ratio 4:1

Any deviation from the recommended design parameters may impact granulation. (See Figure 9)
Granulation Speed
Granulator speed is critical for good granulation, the granulator should be designed for 40% of critical speed, this will assure the material inside the granulator has a cascading action to allow the granule to freely fall inside the granulator and produce good quality material. If the speed is too slow the material will slide inside the granulator or start to roll but it may not be cascading which would result in poor granulation. The critical speed is defined as the rotational speed at which the centrifugal force equals or exceeds the gravitational force and the material no longer rolls or slides. (See Figure 10)

![Figure 10: Granulator Speed](image)

Slurry Nozzles
The location and type of slurry nozzles used is critical to good granulation. Slurry nozzles should be designed to produce even flow pattern on the granulation bed and be uniformly distributed along the length of the granulator. This will also help reduce ammonia losses from the granulator. (See Figure 11)

![Figure 11: Slurry Nozzles](image)

Plugged nozzles will cause irregular spray pattern and poor distribution of slurry resulting in poor granulation. If the nozzle angle is not correct it can cause buildup on supports inside the granulator, resulting in excess downtime or reversing of the granulator, again causing poor granulation. The correct number of nozzles and location along the length of the granulator is also important for good granulation.
Ammonia Sparger
The ammonia sparger must provide equal distribution of ammonia; this is accomplished with a careful design of the sparger holes and the distribution chamber (duck bill). The position of the ammonia sparger is also critical as it should be directed downward and opposite the granulation rotation.

A plugged ammonia sparger or poor distribution of ammonia will cause poor granulation. Over ammoniation will result in a dry, dusty, smaller product and increase ammonia losses. Under ammoniation will cause the granulator to run wet and cause an increase in oversize material, may plug chutes and increase buildup in the downstream equipment. (See Figure 12)

![Figure 12: Ammonia Sparger](image)

Screens
Screening is critical to good granulation, critical design considerations are screen mesh size, screen loading feed rate/screen area, vibration, slope, feed distribution on screens and maintenance.

Operator Interface
A good operator is essential to good granulation, as the operator will monitor many of the parameters discussed in this paper and make the appropriate adjustments based on feed acid lab results and observations made in the plant.

Feed Acid Chemical Composition
The feed acid used by the granulation plant has a significant impact on granulation, impurities such as; iron, aluminum and magnesium will affect the granulation. For Example; too much magnesium will cause the granulation to be dusty and increase the amount of fines. Iron and aluminum can improve granulation by acting as seed crystals that start the initial granulation. The amount of solids in the feed acid also has a significant impact on granulation. If the solids in the feed acid are too low there will not be enough seed crystals to start the granulation mechanism and granulation will be poor. Each plant will have its own unique chemistry but optimizing these parameters will allow for improved granulation.
**Conclusion**
Using the state of the art granulation techniques will produce the best granular product meeting high quality standards. The granulation plants can consistently meet the industry quality standard, typically 2 by 4 mm granules between 93-95% with SGN 225 – 300 and UI 50 – 60.

**References**