AMMONIATION-GRANULATION RESEARCH

Laboratory Design and Operation of Apparatus for Ammoniation-Granulation of Triple Superphosphate

S. A. GIDDINGS and D. DeLAPP

Plant Science Section, Agricultural Division, American Cyanamid Co., Stamford, Conn.

A laboratory ammoniator–granulator has been built and tested which can be used to study ammoniation and granulation on 1000-gram samples under a wide range of operating conditions. The apparatus was found to give reproducible results and to reflect the granulating tendencies of triple superphosphate used in mixed fertilizer production on a plant scale. It is suggested that the performance of fertilizer mixtures on a large scale can be predicted and, hence, reduce the materials and expense involved in conducting trials in the plant.

THE ESTABLISHED physical form of solid fertilizers supplied in the Middle West is granular. The common sense advantages it provides to the farmer easily explain why acceptance is widespread. To the manufacturer of fertilizer, on the other hand, it has intensified the need for new production methods and additional requirements for raw materials. A good review of current methods of granulation used in the industry is given by Hignett and Slack (*7*).

The efficiency of an ammoniation–granulation process is affected by temperature, quality characteristics of raw materials, quantity and chemical composition of liquid phase, equipment design, and rolling speed. Hence, with this long list of variables it is not surprising that difficulties have arisen in trying to manufacture a product of an exact N–P_2N_5–K_2N composition, such as a 5–20–20 granular fertilizer, and still maintain high throughput, low raw materials, and operating costs. These problems are overcome in fertilizer plants throughout the country, but it is still fair to say that a sizable component of the art and knowledge based on operating experience contribute to profitable granulation.

Several studies on small-scale equipment established a fundamental understanding of the important factors in the granulation process (*1, 4, 8, 9*). The purpose of the apparatus described here is for the study of these factors during simultaneous ammoniation–granulation, analogous to plant operation in Tennessee Valley Authority–type equipment. Reproducible numerical values of granulation and ammonia absorption efficiencies have been obtained on 1000-gram charges of triple superphosphate. The expense of large-scale trials could probably be reduced from initial results of trials in this equipment.

The fundamental components of a suitable laboratory scale apparatus include a small, rolling drum, a means of accurately adding a predetermined quantity of gaseous ammonia to a bed of solid fertilizer raw materials, a temperature recorder, and provision for drying the granules once formed. The apparatus constructed for this study embodies important advances over earlier granulators, such as easily adjustable speed of rotation, opportunity for visual observation of the rolling bed, variable placement of the ammonia sparger, control over the addition rate of ammonia, relatively small quantity of

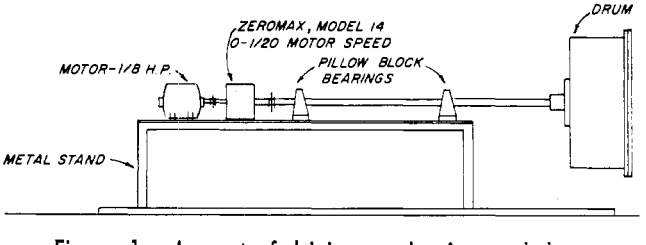

Figure 1. Layout of driving mechanism and drum

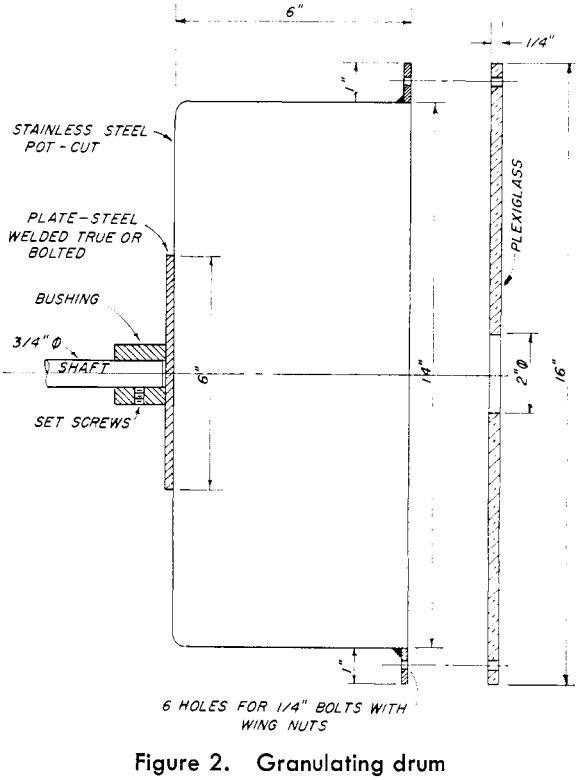

Figure 2. Granulating drum

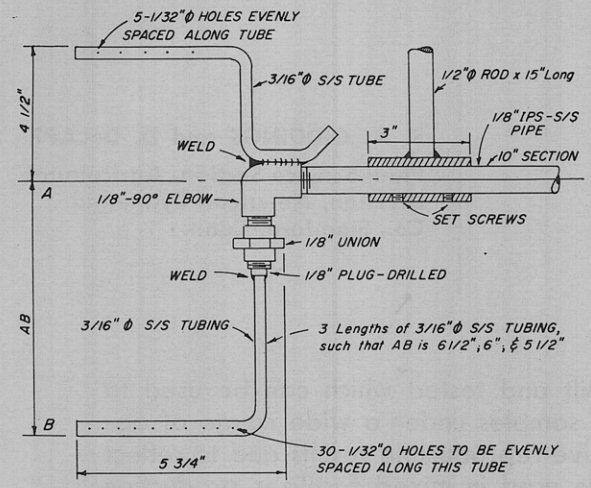

Figure 3. Ammonia and water distributor

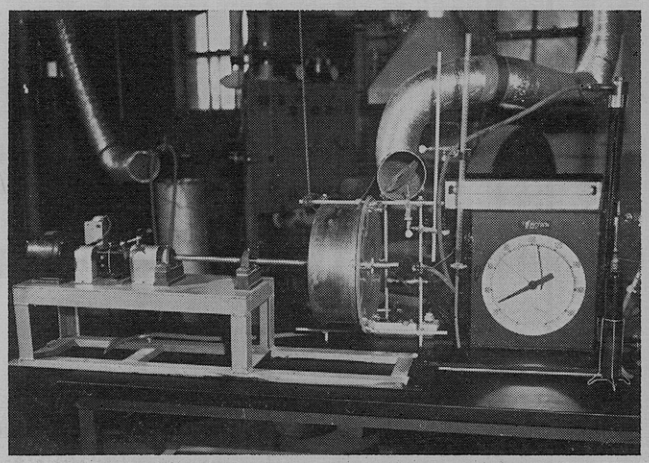

Figure 4. Apparatus

sample required, and particularly, convenience in loading, unloading, and cleaning up for the next run.

Description of the Granulator

A small unit was constructed to meet the needs as outlined. Schematic drawings of the driving mechanism and drum, the details of the drum itself, and the distribution equipment are depicted in Figures 1 to 3. With the motor and variable speed reducer, the speed of rotation of the drum could be varied between 0 and 60 r.p.m. A picture of the completed apparatus set in position for a run appears in Figure 4. It will be noted from the picture that a lecture bottle of ammonia is connected through a rotameter to the ammonia sparger. Also, a 100-ml. buret is positioned above the distributor for adding measured amounts of water to the rolling bed through the water distributor shown in Figure 3.

Operation of the Granulator

Sample Preparation. A representative portion of the triple superphosphate or fertilizer mixture to be tested must be used because granulation is markedly affected by particle size distribution. Large samples were quartered and finally riffled until 1000 grams were separated for the test run. The triple superphosphate samples were analyzed for total P_2O_5, available P_2O_5, citrate-insoluble P_2O_5, free acid as P_2O_5, moisture, particle size distribution, and in some cases, porosity by mercury intrusion measurements. For studies involving the investigation of granulation of specific particle sizes or controlled ratios of the various fractions, the materials were first sieved and then recombined, if necessary, to give direct comparisons of the granulating tendencies of various samples. The moisture was adjusted to equal levels when highest accuracy was desired. This was readily achieved by addition and then equilibration in a closed container, or by removing a predetermined amount of moisture in an oven.

Anhydrous Ammonia Supply. A 1.5 × 10 inch empty ammonia lecture bottle was loaded with the calculated amount of anhydrous ammonia needed for a run. The lecture bottle was connected to the pressure gage of the main cylinder with steel fittings and then placed in a Dewar flask containing acetone and dry ice to condense sufficient ammonia in the tared bottle. The lecture bottle was disconnected and weighed at 5-minute intervals until a few grams excess had been collected. With a little experience and using identical conditions, the time of addition was approximated closely for successive trials. Any excess was vented into a well ventilated hood until the required quantity of ammonia remained in the bottle. The lecture bottle was never filled more than three quarters because of the danger of expansion of the liquid and subsequent rupture. The gas space remaining prevented the pressure from rising above the critical pressure of ammonia. For granulation studies, a level of 4 pounds of ammonia per 20 pounds of P_2O_5 was used since this value approaches the upper level which can be used without excessively lowering ammoniation efficiency (*5*, *8*).

Procedure for Ammoniating and Granulating Triple Superphosphate. The variable speed drive was adjusted and tested for the revolutions per minute desired in the run. Ordinarily, a speed of 35 r.p.m. was used corresponding to 50% of the critical speed, which Brook (*1*) had demonstrated to yield optimum granulation. (Critical speed is the number of revolutions per minute which enables solid material in the drum to maintain its position relative to the drum by centrifugal force; it is equal to $\frac{76.5}{\sqrt{d}}$ where d is the diameter in feet of the drum.) The drum was then charged, the sparger assembly inserted, and the face plate attached. Next, the assembly was clamped into position for distributing water on the surface of the rolling bed at a location where the charge was tumbling rapidly down in a direction opposite to the motion of the drum.

The temperature sensing element, just protruding through a suitably bent, narrow stainless steel tube, was supported on the sparger assembly so that the temperature was measured 1 inch behind the ammonia distributor and in the center of the material at this point. It will be noted that the direction of flow of ammonia is away from the zone in which the temperature is being measured. The ammonia bottle, surrounded by water at 70° C. to prevent evaporative chilling and reduction in flow, was connected through the rotameter (0.4 to 2.5 cu. feet per minute of ammonia) and a 100-ml. buret was connected to the water distributor by a piece of rubber tubing. If a seesaw motion of the solids occurred in the drum during water addition, a few raps on the drum with a hammer handle or slight wetting of the internal drum surface were usually sufficient to initiate good bed action, although some materials were rather recalcitrant. Suffice to say that the water should be distributed evenly rather than to overwet portions of the solids. The mixture was blended thoroughly for 1 minute, at which time the distributor assembly was rotated until the ammonia sparger was under the bed at approximately the 7 o'clock position. Ammonia flow was commenced simultaneously with, or 1 to 2 seconds before, the rotation of the ammonia distributor into the material. The flow rate was predetermined to give the desired total ad-

dition time. Three minutes (±10 seconds) were chosen as the standard time being both practical in the laboratory and corresponding to conditions in large plants (*2, 3*). The rotameter reading cannot be used to measure the absolute quantity of ammonia gas added as pressure in the sparger system varies between one run and another.

The temperature rose quickly (approximately 1 minute) from ambient to 100° to 110° C. and remained steady due to the cooling effect of water evaporation. After ammonia addition, the distributor was rotated free of the bed and the material allowed to roll for 2 minutes. The distributor was replaced under the bed and air introduced at a rate of 2.2 to 2.4 cu. feet per minute for 10 minutes

Calculation of Results

Determination of Degree of Granulation. A particle size distribution was made on half the sample using Tyler 6, 10, 20 and 35 nested screens by rotapping for 1 minute, a period found to be adequate for granulated material. The per cent onsize was chosen as 2/3 the +6 fraction plus the −6 + 20 fraction. Onsize has been considered by earlier workers (*6, 10, 11*) as ranging from −6 + 16 to −6 + 28. Examination of results, reported in these articles, of crushing the oversize and its effect on the final onsize showed in all cases that approximately two thirds of the oversize was converted to onsize material. The degree of granulation for the run is then defined as the per cent onsize after granultion minus the per cent onsize before granulation.

Determination of Ammoniation Efficiency. A representative sample of the product was analyzed for total P_2O_5 and total NH_3 by standard procedures. The ammoniation efficiency is then

$$\frac{\text{pounds } NH_3 \text{ found/pound } P_2O_5 \times 100}{\text{pounds } NH_3 \text{ charged/pound } P_2O_5}$$

Alternately the efficiency may be calculated, provided that the total weight of material remaining in the drum after the run was measured precisely. The only analytical determination required is the total NH_3 in the product. From the initial amount of NH_3 added, the ammoniation efficiency is then

$$\frac{\%\ NH_3 \text{ found} \times \text{final weight}}{\text{grams } NH_3 \text{ charged}}$$

Reproducibility

Table I lists granulation and ammoniation results of two representative samples of triple superphosphate, each run three times to show the reproducibility that can be expected.

Correlation of Laboratory and Plant Trials

Facilities to carry out or supervise granulation trials on a full scale using materials previously evaluated in the laboratory apparatus were unfortunately unavailable. However, operating data taken from a plant trial of granular (5–20–20) fertilizer manufacturer were supplied by the technical and supervisory personnel who conducted the test. Two different sources of triple superphosphate were used in the formulation, while all of the other raw materials were common to both runs. One sample was observed to promote efficient granulation, while the other required large quantities of water in order to prevent undergranulation and excessive recycle of fines. Data listed in Table II were obtained at steady state under similar operating conditions.

A laboratory ammoniation–granulation was also made on two samples of triple superphosphate. The speed of rotation, time of rolling, moisture content, etc., were controlled at the same level for each run. To ensure further a a precise comparison of the inherent granulating tendencies of the two samples without influence of the small particle size variation between them, each 1000-gram charge was prepared so that their particle size distributions were identical.

Table II demonstrates the correlation

Table I. Experimental Results

Run No.	Particle Size Distribution after Granulation +6	−6 +10	−10 +20	−20	% Final Onsize	% Ammonia Absorption
Sample I						
1	12.1	15.8	25.6	46.4	49.4	96
2	17.6	12.4	23.8	46.1	47.8	98
3	10.9	16.6	24.0	48.4	47.8	98
Sample II						
1	8.4	36.4	24.7	30.6	66.7	95
2	6.9	36.0	25.8	31.7	66.4	95
3	5.7	35.8	24.3	34.2	63.9	98

Table II. Plant Manufacture of 5–20–20 Fertilizer

Formulation

Material	Pounds/Ton
Anhydrous ammonia	73
Nitrogen solution 49 (34-60-0)	90
Normal superphosphate	380
Triple superphosphate	706
Muriate of potash	662
Sulfuric acid (93%)	140
Water	140

Particle Size of Triple Superphosphate

Sample	Size −10	−35	−65
A	89.1	56.2	26.8
B	87.5	53.4	24.6

Final Product Particle Size Exit Cooler

Conditions	Size +6	−6 +10	−10 +14	−14 +20	−20 +24	−24	Field Rating
Product when *A* used as triple superphosphate	8.4	16.5	13.8	25.8	16.7	18.8	Good
Product when *B* used as triple superphosphate	4.4	5.9	5.6	19.8	27.3	37.0	Poor

Laboratory Granulation Results

Sample	Initial Particle Size Distribution (Both Samples) +5	−6 +10	−10 +14	−14 +20	−20 +35	−35 +65	−65 +100	−100	% 2/3 (+6) + (−6 +20) Degree of Granulation
	1.2	8.7	4.3	7.1	21.6	32.3	6.4	18.7	20.9
A	Particle Size Distribution after Granulation 9.2	24.1	29.8	23.0	13.6	0.3	...	...	83.3 / 62.1
B	12.8	19.0	12.2	14.7	36.2	5.0	...	...	54.4 / 33.5

between plant operation and laboratory-scale apparatus. There have been more than 250 runs made in our equipment in an endeavor to understand better the nature of the materials to be ammoniated and granulated, and the factors affecting granulation and ammoniation. These results will be reported at a later date.

Conclusion

The laboratory ammoniator–granulator which has been built and tested permits determination of granulation efficiency and ammonia absorption under controlled conditions with small quantities of raw materials.

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FERTILIZER TECHNOLOGY

Application of Slurry-Type Processes in the TVA Ammoniator-Granulator

R. D. YOUNG, G. C. HICKS, and C. H. DAVIS

Tennessee Valley Authority, Wilson Dam, Ala.

Pilot-plant studies were made of several processes for the production of granular, high-analysis fertilizers in which reaction slurries or liquids comprised a major proportion of the feed to a TVA-type ammoniator-granulator. Processing was characterized by relatively high recycle ratios (1:1 to 5:1), since recycle was used as the primary control of granulation. Data are presented to show the effects of process variables on recycle requirements, flexibility of control of operation, and quality of granules. Variables studied included formulations, methods of slurry distribution, size and temperature of recycle, size range of granulator discharge, and special drying, crushing, and screening procedures.

Since its development, about 10 years ago, the Tennessee Valley Authority (TVA) continuous ammoniator has met wide acceptance in the fertilizer industry. There are now more than 150 ammoniators of this type in use in plants in 37 states. It is estimated that over two thirds of the granular fertilizer used in the United States in the past few years was produced in these units.

More recently, pilot-plant work with the TVA-type ammoniator has shown that it can be used to advantage as an acidulating drum in the production of superphosphates (*4*) and for processing slurries of fertilizer materials (*2*). There is at present limited commercial application of these processes.

This article discusses the use of the ammoniator in slurry-type processes, which are of interest because of the generally lower cost of raw materials and the variety of higher analysis grades that can be produced. The material covered does not entail a complete description of any one process, but includes general considerations based on observations and data obtained in pilot-plant work on several slurry-type processes.

Pilot-plant studies that form the basis for this paper include the following.

Utilization of Calcium Metaphosphate. In this work a slurry of hydrolyzed calcium metaphosphate was ammoniated and granulated with other fertilizer materials to produce a variety of grades of granular fertilizers (*5*).

Nitric Phosphates. Extraction slurry of nitric and sulfuric or phosphoric acids and phosphate rock was ammoniated and granulated with other fertilizer materials in the continuous ammoniator (*2*).

Preneutralization Studies. This work included prereaction of sulfuric acid, wet-process and furnace phosphoric acids, and nitric acid with ammonia and/or nitrogen solutions in an open tank followed by completion of ammoniation and granulation with other fertilizer materials in the TVA-type ammoniator-granulator. This would be considered as a partial slurry-type operation, since solid fertilizer materials usually comprise a considerable portion of the formulations.

Production of Granular Diammonium Phosphate. This process recently developed by TVA utilizes a preneutralizer and continuous ammoniator. Excess ammonia fed to the drum to produce diammonium phosphate is recovered by scrubbing the exhaust gases with the phosphoric acid used in the process. Straight diammonium phosphate as well as several N–P–K grades can be produced (*3*).

Equipment Requirements and Processing Characteristics

Conventional ammoniation-granulation plants require additional equipment for utilizing slurry-type processes. Changes in and additions to the plant are comparatively minor for use of a preneutralizer and are at present included in several plants. For separate process application, such as the production of nitric phosphates and granular diammonium phosphate, more equipment is required. In the nitric phosphate process (*2*), a two-stage extraction unit is required. Auxiliary equipment for feeding phosphate rock and acid and facilities for storage of the nitric acid also are required. The process for granular diammonium phosphate (*3*) requires a preneutralizer and a scrubber for recovery of ammonia as the main additional equipment.