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FERTILIZER TECHNOLOGY

Granulation Characteristics of a 5–4–12 (5–10–15) Fertilizer Containing Potassium Nitrate

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Factors influencing the granulation of a mixed fertilizer containing potassium nitrate have been studied. Kilogram batches of a 5–4–12 (5–10–15) mixed fertilizer containing potassium nitrate were granulated at different feed moisture levels and at inlet gas temperatures ranging from 405° to 575° F. Optimum conditions of moisture in the feed and inlet gas temperature were determined for maximum yield of the granular product and uniform distribution of the three major nutrient elements (N, P, K) in the various product size fractions. Similar experiments were performed with a 2–4–12 (2–10–15) mixed fertilizer containing potassium chloride. This grade represents a simple substitution of KCl for KNO_3 so physical changes due to KNO_3 could be more closely compared. A comparative study has been made of the results obtained with the two mixtures. The experiments were based on a statistical central composite rotable design, and the results were statistically analyzed. An attempt has been made to relate mathematically the yield and total absolute deviation in nutrient analysis between product and feed with moisture in a feed and temperature of granulation.

Potassium nitrate has a high agronomic value as a source of both nitrogen and potash, but high cost has been the chief reason for its limited use as fertilizer. The new production facilities of Southwest Potash Corp. for fertilizer-grade potassium nitrate, 13% N, 36.5% K (44% K_2O), may make this material economically attractive. This has stimulated considerable interest in the properties of potassium nitrate with respect to its behavior in mixed fertilizers during processing and its effect on the physical quality of the resulting products.

Previous Work

During the past few years, much work has been done on the granulation characteristics of mixed fertilizers. Pilot plant studies conducted at TVA (*3*) showed that in production of very low nitrogen grades [e.g., 3–5–9 (3–12–12) or 4–6–13 (4–16–16)] the amount of moisture required for granulation of the charge material was high (14 to 16%), and the products were rather wet. Smith (*4*), in the study of temperature and moisture relationships in granulation, noted that the utilization of ammonia as anhydrous or nitrogen solutions reduced not only the cost of nitrogen but also the free water content. A high salt solution phase contributed to rapid crystallization and therefore aided formulation of granules during agglomeration. These grades contained potassium nitrate as the source of potassium. Recently, Hardesty and his coworkers, working with grades containing potassium nitrate, noted that slightly less moisture was required for agglomeration with potassium nitrate mixtures (*2*), but they produced a "popped-corn" shape of granules which effloresced on drying. Comparison of the yield and homogeneity of the product with mixtures that contained potassium nitrate and potassium chloride was not reported.

Experimental Work

Experiments were performed to study the effect of moisture in the feed and inlet gas temperature on the yield and the distribution of plant nutrients in the various product size fractions. Two

Figure 1. Photograph of the ammoniator-mixer disassembled

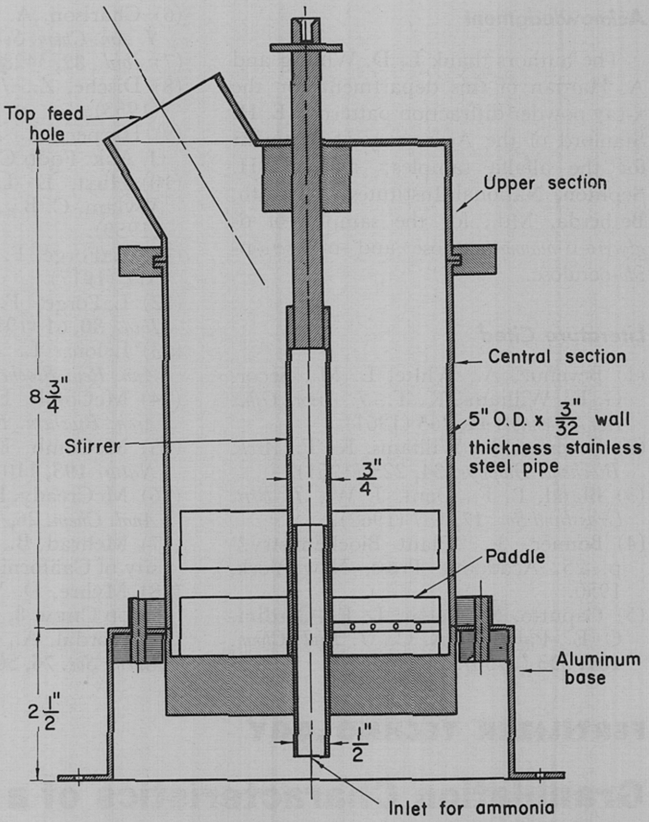

Figure 2. Sectional drawing of ammoniator-mixer

grades were prepared—5–4–12 (5–10–15) tobacco fertilizer with potassium nitrate as the major source of potassium, and 2–4–12 (2–10–15) mixed fertilizer with potassium chloride as the major source of potassium (Table I). The solid raw materials (all crushed to −20 mesh) were mixed in a batch reactor, and then reacted with sulfuric acid and ammonia. The required moisture content was obtained by addition of predetermined amounts of water to the feed mixtures.

The batch reactor was constructed from a section of 5-inch o.d., type 304, stainless steel tube with a wall thickness of $^3/_{32}$ inch (see Figures 1 and 2). A stainless steel stirrer, with a hollow shaft and four paddles, was used to mix the ingredients and to distribute ammonia.

The feed materials were screened and stored in individual bins. As needed, the required quantities were removed from the bins and dry mixed in the mixer. The water, sulfuric acid, and anhydrous ammonia were added one at a time to the mixture and mixing was continued for a predetermined time. Before transferring the mixture to the granulator dryer, duplicate samples were removed for analysis.

The mixtures were granulated in a bench-scale Roto-Louvre dryer 13 inches in diameter and $3^1/_8$ inches long (Figure 3). The dryer speed was 7.4 r.p.m. The product was dried, screened, and analyzed for nitrogen, phosphorus, and potassium.

Experiments on both grades were statistically designed (*1*). The two major factors, per cent moisture in the feed and average inlet gas temperature, were varied over a reasonable range. Preliminary experiments showed that even a variation of 0.5% moisture in the feed affected the yield of granules appreciably with the 5–4–12 (5–10–15) grade, while 1.5% moisture variation was necessary to get an approximately equivalent effect on the yield of granules with the 2–4–12 (2–10–15) mixture. The experiments with the 5–4–12 (5–10–15) grade were performed randomly according to the design in Figure 4, and those with the 2–4–12 (2–10–15) according to the design in Figure 5.

Table I. Formulation of 5–4–12 (5–10–15) and 2–4–12 (2–10–15) Mixed Fertilizers

Material	Analysis				Formula, Lb. / Ton	
	N	P(P_2O_5)	K(K_2O)	Mg(MgO)	5–4–12 (5–10–15)	2–4–12 (2–10–15)
Superphosphate	0	8.95(20.5)	0	0	1020	1020
Anhydrous ammonia	82.2	0	0	0	52	52
Sul-Po-mag	0	0	18.1(21.7)	10.8(18)	222	222
Potassium nitrate	13.2	0	36.1(43.4)	0	460	0
Potassium chloride	0	0	50.0(60.2)	0	75	425
Sulfuric acid	0	0	0	0	50	50
Filler (Sand)	0	0	0	0	171	281
				Total	2050	2050

Two mathematical models were set up—model I, relating the per cent yield with per cent moisture in the feed and temperature of the inlet gas, and model II, relating the total absolute deviation of the three nutients N, P, and K in the product from that in feed with per cent moisture in feed and temperature of the inlet gas. The total absolute deviation was the statistic which expressed the variation of the plant nutrients between the feed and the granular product. It was estimated for every experimental run by summing up the individual absolute differences for N, P, and K between feed and −6 + 20 granular product.

$$Y = b_0 + b_1x_1 + b_2x_2 + b_{11}x_1^2 + b_{22}x_2^2 + b_{12}x_1x_2$$

Model I:

Y = % Yield (−6+20 mesh granules)
x_1 = Average inlet gas temperature, ° F.
x_2 = % Moisture in feed (dry basis).

Model II:

Y = Total absolute deviation of N–P–K in product from that in feed.
x_1 = Average inlet gas temperature, ° F.
x_2 = % Moisture in feed (dry basis)

The regression coefficients b_0, b_1, b_2, b_{11}, b_{22}, b_{12} of these models were determined from the experimental results.

Discussion

The experimental results with both the grades are shown in Tables II, III, IV,

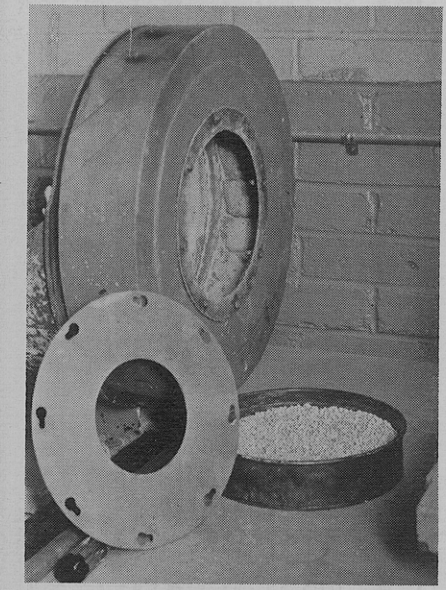

Figure 3. Photograph of granulator-dryer and granulated product

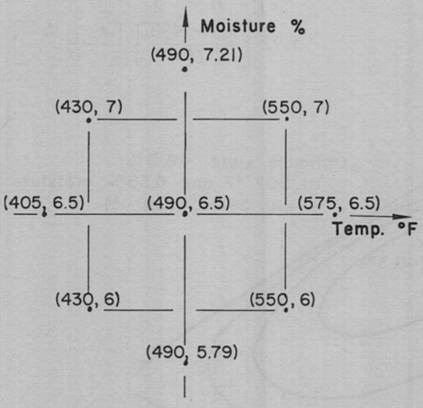

Figure 4. Schematic of central composite rotatable design for the 5–4–12 (5–10–15) grade containing potassium nitrate

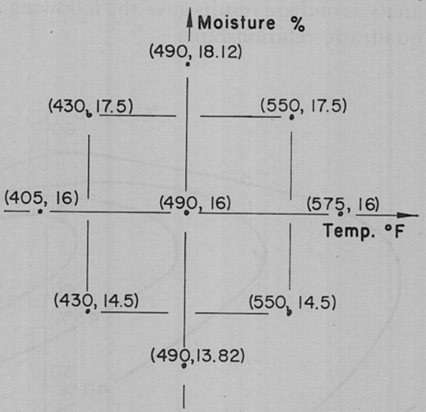

Figure 5. Schematic of central composite rotatable design for the 2–4–12 (2–10–15) grade containing potassium chloride

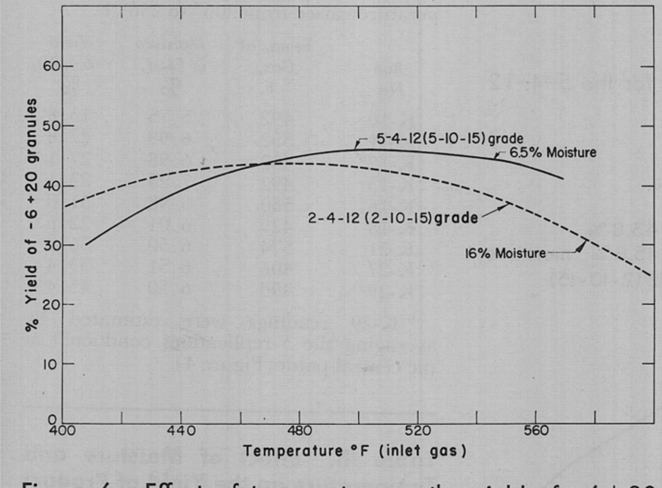

Figure 6. Effect of temperature on the yield of $-6+20$ product size fraction for both fertilizer grades at their central moisture levels

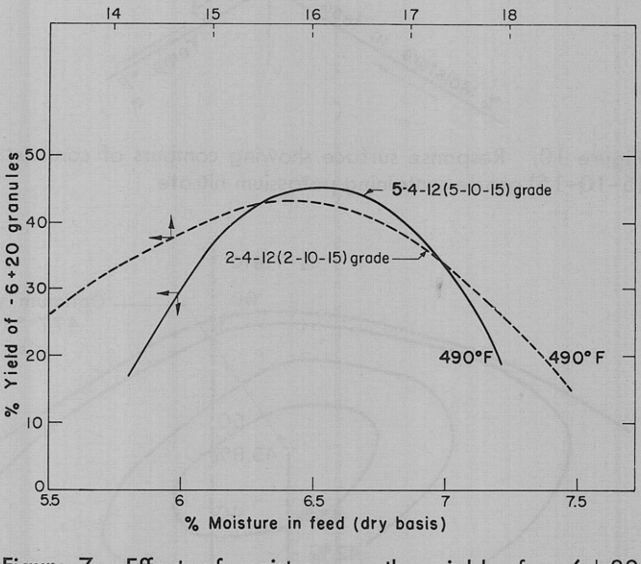

Figure 7. Effect of moisture on the yield of $-6+20$ product size fraction for both fertilizer grades at their central temperature level

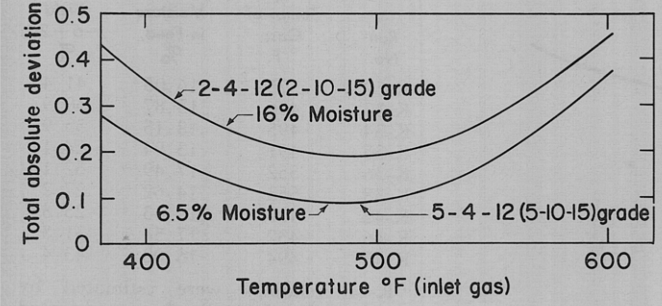

Figure 8. Effect of temperature on the total absolute deviation for both fertilizer grades at their central moisture levels

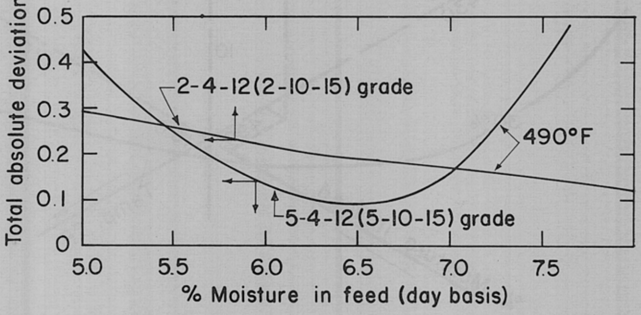

Figure 9. Effect of moisture on the total absolute deviation for both fertilizer grades at their central temperature level

and V. Tables II and III show the effect of moisture and temperature on the yield of granules, and Tables IV and V present the effect of moisture and temperature on the total absolute deviation in the product. The statistical analysis of these results gave the following quadratic relationships:

Model I:

$$Y = 45.40 + 3.36\left[\frac{x_1 - 490}{60}\right] + 1.8\left[\frac{x_2 - 6.5}{0.5}\right] - 5.17\left[\frac{x_1 - 490}{60}\right]^2 - 13.37\left[\frac{x_2 - 6.5}{0.5}\right]^2 - 1.92\left[\frac{x_1 - 490}{60}\right]\left[\frac{x_2 - 6.5}{0.5}\right]$$

with the 5–4–12 (5–10–15) grade.

$$Y = 43.36 - 2.10\left[\frac{x_1 - 490}{60}\right] - 3.05\left[\frac{x_2 - 16.0}{1.5}\right] - 4.46\left[\frac{x_1 - 490}{60}\right]^2 - 8.04\left[\frac{x_2 - 16.0}{1.5}\right]^2 - 0.20\left[\frac{x_1 - 490}{60}\right]\left[\frac{x_2 - 16.0}{1.5}\right]$$

with the 2–4–12 (2–10–15) grade.

Model II:

$$Y = 0.0960 + 0.0175\left[\frac{x_1 - 490}{60}\right] + 0.0125\left[\frac{x_2 - 6.5}{0.5}\right] + 0.0664\left[\frac{x_1 - 490}{60}\right]^2 + 0.0464\left[\frac{x_2 - 6.5}{0.5}\right]^2 - 0.0575\left[\frac{x_1 - 490}{60}\right]\left[\frac{x_2 - 6.5}{0.5}\right]$$

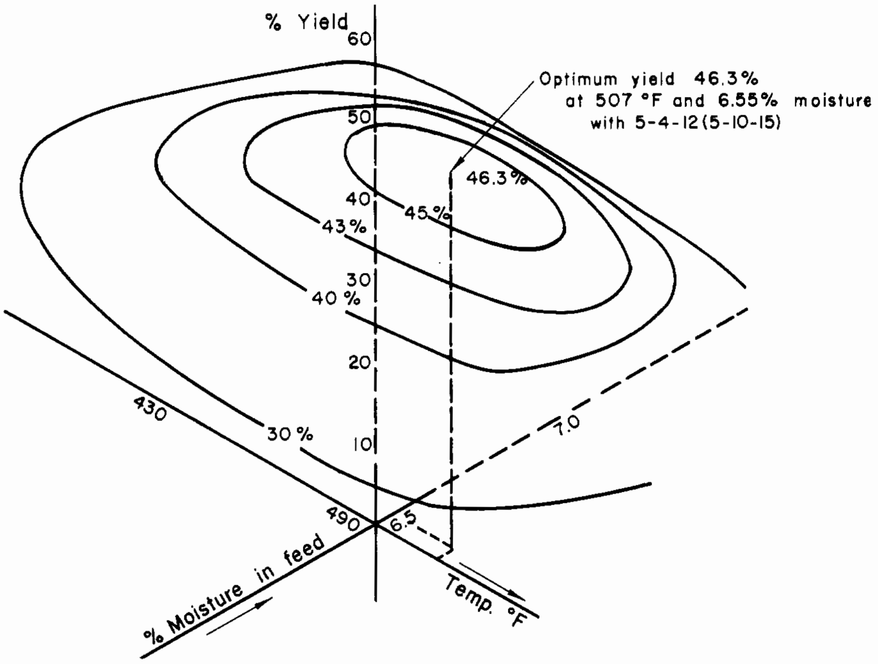

Figure 10. Response surface showing contours of constant yield for the 5–4–12 (5–10–15) grade containing potassium nitrate

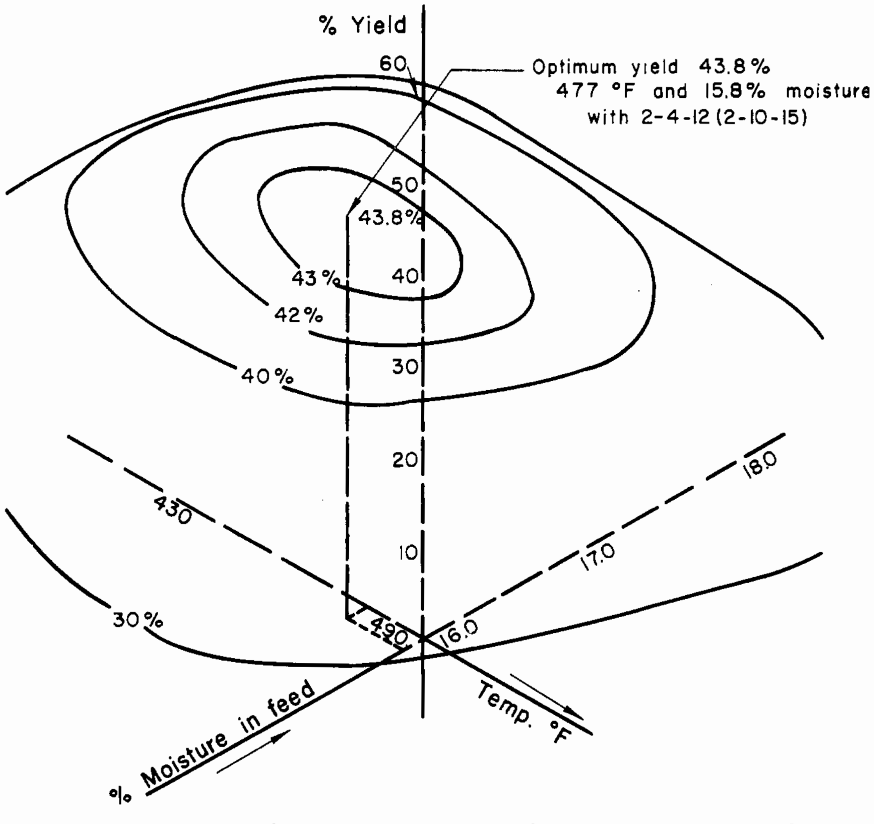

Figure 11. Response surface showing contours of constant yield for the 2–4–12 (2–10–15) grade containing potash chloride

Table II. Effect of Moisture and Temperature on the Yield of Product for the 5–4–12 (5–10–15) Grade Containing Potassium Nitrate

Drying time was 20 minutes.

Moisture in the product varied from 0.38% to 0.92%. Average product temperature varied from 203° to 256° F.

Run No.	Temp. of Gas, ° F.	Moisture in Feed, %	Yield −6+20, %
K-10	492	5.75	13.8
K-11	552	6.99	27.8
K-12	432	6.98	27.1
K-13	492	7.23	23.1
K-16	550	5.98	31.0
K-18	429	6.01	22.6
K-21	574	6.50	36.2
K-27	406	6.51	33.4
K-29[a]	490	6.50	45.4

[a] K-29 readings were estimated by averaging the 5 replications conducted at the central point (Figure 4).

Table III. Effect of Moisture and Temperature on the Yield of Product for the 2–4–12 (2–10–15) Grade Containing Potassium Chloride

Drying time was 45 minutes.

Moisture in the product varied from 0.29% to 0.92%.

Average product temperature varied from 194° to 249° F.

Run No.	Temp. of Gas, ° F.	Moisture in Feed, %	Yield −6+20, %
K-32	575	16.05	41.4
K-33	406	15.87	30.9
K-34	495	18.15	33.9
K-35	491	13.94	24.1
K-36	552	17.49	32.1
K-38	553	14.62	27.2
K-42	430	14.53	25.8
K-46	432	17.54	31.5
K-47[a]	494	16.08	43.4

[a] K-47 readings were estimated by averaging the 5 replications conducted at the central point (Figure 5).

Table IV. Effect of Moisture and Temperature on Total Absolute Deviation of Plant Nutrients for 5–4–12 (5–10–15) Grade Containing Potassium Nitrate

Run No.	Temp. of Gas, °F.	Moisture in Feed, %	Nutrient Analysis of −6 + 20 Size Product					Abs. Dev. of Nutrients between −6 + 20 Size Product and Feed			Total Abs. Dev.
			N	P[a]	(P_2O_5)[a]	K	(K_2O)	N	P	K	
K-10	492	5.75	5.31	5.92	(13.57)	12.58	(15.15)	0.08	0.01	0.08	0.17
K-11	552	6.99	5.45	5.98	(13.71)	12.79	(15.40)	0.10	0.04	0.08	0.22
K-12	432	6.98	5.79	5.92	(13.58)	12.65	(15.25)	0.15	0.05	0.08	0.28
K-13	492	7.23	5.59	5.95	(13.64)	12.44	(15.00)	0.11	0.03	0.04	0.18
K-16	550	5.98	5.38	6.00	(13.74)	12.69	(15.30)	0.14	0.05	0.10	0.31
K-18	629	6.01	5.51	5.86	(13.43)	12.65	(15.25)	0.06	0.00	0.08	0.14
K-21	574	6.50	5.23	5.90	(13.52)	12.69	(15.30)	0.12	0.05	0.04	0.21
K-27	406	6.51	5.60	5.92	(13.69)	12.58	(15.15)	0.14	0.01	0.04	0.19
K-29[b]	490	6.50	5.53	5.87	(13.48)	12.68	(15.27)	0.02	0.01	0.06	0.09

[a] Total.

[b] K-29 readings were estimated by averaging the five replications conducted at the central point (Figure 4).

Table V. Effect of Moisture and Temperature on Total Absolute Deviation of Plant Nutrients for 2–4–12 (2–10–15) Grade Containing Potassium Chloride

Run No.	Temp. of Gas, °F.	Moisture in Feed, %	Nutrient Analysis of −6 + 20 Size Product					Abs. Dev. of Nutrients Between −6 + 20 Size Product and Feed			Total Abs. Dev.
			N	P[a]	(P_2O_5)[a]	K	(K_2O)	N	P	K	
K-32	575	16.05	2.26	5.83	(13.38)	12.81	(15.50)	0.21	0.03	0.12	0.36
K-33	406	15.87	2.48	5.82	(13.37)	13.23	(15.85)	0.01	0.03	0.30	0.34
K-34	495	18.15	2.32	5.94	(13.62)	12.92	(15.60)	0.05	0.00	0.04	0.09
K-35	491	13.94	2.41	5.91	(13.51)	12.98	(15.65)	0.10	0.02	0.17	0.29
K-36	552	17.49	2.36	5.96	(13.63)	12.92	(15.60)	0.08	0.02	0.12	0.22
K-38	553	16.62	2.39	5.84	(13.39)	12.81	(15.50)	0.24	0.03	0.00	0.27
K-42	430	16.53	2.39	5.86	(13.41)	13.19	(15.80)	0.09	0.02	0.12	0.23
K-46	432	17.54	2.47	5.87	(13.46)	12.92	(15.60)	0.07	0.02	0.17	0.26
K-47[b]	494	16.08	2.45	5.92	(13.57)	12.79	(15.45)	0.05	0.02	0.12	0.19

[a] Total.

[b] K-47 readings were estimated by averaging the five replications conducted at the central point (Figure 5).

with the 5–4–12 (5–10–15) grade.

$$Y = 0.1900 + 0.0038\left[\frac{x_1 - 490}{60}\right] - 0.0380\left[\frac{x_2 - 16.0}{1.5}\right] + 0.0737\left[\frac{x_1 - 490}{60}\right] + 0.0063\left[\frac{x_2 - 16.0}{1.5}\right]^2 - 0.0200\left[\frac{x_1 - 490}{60}\right]\left[\frac{x_2 - 16.0}{1.5}\right]$$

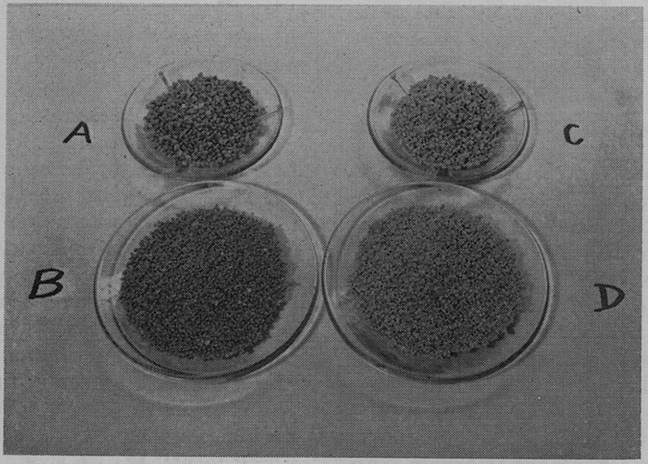

Figure 12. Photographs of granular products

A: −6+8 size fraction of the 5–4–12 (5–10–15) grade containing potassium nitrate
B: −8+16 size fraction of the 5–4–12 (5–10–15) grade containing potassium nitrate
C: −6+8 size fraction of the 2–4–12 (2–10–15) grade containing potassium chloride
D: −8+16 size fraction of the 2–4–12 (2–10–15) grade containing potassium chloride

with the 2–4–12 (2–10–15) grade.

The effect of temperature on the yield with the two mixtures may be shown graphically (Figure 6). The yield was generally higher with the 5–4–12 (5–10–15) formulation. Because the tendency to form large lumps was greater at higher temperatures with the 2–4–12 (2–10–15) mixture, the yield decreased more rapidly.

Moisture was more critical in the 5–4–12 (5–10–15) than in the 2–4–12 (2–10–15), and affected the yield to a greater extent (Figure 7).

The effect of temperature on the total absolute deviation with the two mixtures may be shown graphically (Figure 8). The total absolute deviation was much lower with the 5–4–12 (5–10–15) mixture than with the 2–4–12 (2–10–15) mixture, showing the product to be more homogeneous with mixtures containing potassium nitrate.

With the increase of moisture in the feed, the total absolute deviation decreased and, after attaining a minimum, started increasing with the 5–4–12 (5–10–15) mixture. With the 2–4–12 (2–10–15) it continued to decrease; but was considerably higher at the optimum yield condition than with the 5–4–12 (5–10–15) mixture (Figure 9).

Three-dimensional views showing the effect of temperature and moisture on the yield of product are shown in Figures 10 and 11. The lines show contours of constant yield. The distance between the adjacent contour lines parallel to temperature axis is greater for the 2–4–12 (2–10–15) mixture than for 5–4–12 (5–10–15) mixture, indicating that moisture is more critical with mixtures containing nitrate of potash. The optimum yield of 46.3% was noted at 507° F. and 6.55% moisture with the 5–4–12 (5–10–15) mixture, and the optimum yield of 43.8% at 447° F. and 15.8% moisture with the 2–4–12 (2–10–15) formulation.

The granules obtained with the 5–4–12 (5–10–15) mixed fertilizer containing potassium nitrate were more spherical and uniformly shaped than those obtained with the 2–4–12 (2–10–15) mixed fertilizer containing potassium chloride (Figure 12). The 5–4–12 (5–10–15) mixed fertilizer also gave more cohesive, less porous, and tougher granules.

The granule product in both mixtures was relatively more homogeneous than reported in the literature since the raw materials used in this study were of finer mesh size.

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FERTILIZER TECHNOLOGY

A One-Step Continuous Quick-Curing Triple Superphosphate Process Employing Rod Mill Grinding

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An investigation was undertaken to determine the feasibility of a one-step quick-curing continuous process for the manufacture of triple superphosphate. Bench-scale work was carried out in a 1-quart, laboratory ball mill made of stainless steel. The results from this preliminary work indicated that quick-curing triple superphosphate of high conversion could be produced. The process was investigated on pilot-plant scale with successful operation. Completely cured powdered triple superphosphate was obtained within 1 hour.

THE ADVANTAGES of a quick-curing continuous superphosphate process include the following: the product can be shipped directly, thus reducing the required storage facilities and process inventory; operating conditions in the mixing step can be chosen with more flexibility; control of the process can be essentially automatic; uncertainty as to production rates can be eliminated to some extent because a finished product can be produced in a matter of hours, instead of weeks as required by the storage curing process; and there is a possibility of lower operating labor and maintenance requirements and elimination of a final crushing and screening step since finely ground products can be obtained directly.

Previous Work

Many processes (*3*, *5*, *9*, *12*) have been tried for quick-curing of triple superphosphate. None of these processes has been able to eliminate curing time entirely without loss of phosphorus availability. In some processes, both drying and storage curing are employed to shorten the period of curing. A quick-curing process is therefore desirable in which a minimum amount of phosphoric acid is needed to convert a maximum amount of rock into available form without curing. The present paper discusses an investigation of such a process.

The development of a quick-curing, one-step process for normal superphosphate was carried out by Rounsley and Boylan (*14*) and Martinez (*8*), by grinding and drying in a single piece of equipment. This process was extended to the manufacture of triple superphosphate. Essentially the same laboratory and pilot plant equipment were used.

Process Variables

The important process variables which effect the reaction stage and curing stage of the process are acid concentration, temperature, acidulation ratio (acidulation ratio as used here is the weight ratio of acid P_2O_5 to rock P_2O_5), and time. The effects of these variables using grinding in a laboratory ball mill and a pilot plant rod mill were investigated.

The effect of acid concentration is the most important process variable. An acid concentration of about 70% H_3PO_4 has been found most suitable, considering the physical properties of the mixture during the process and the degree of completion of reaction. Higher acid concentrations result in poor conversion, probably due to excessive side reactions. Very low acid concentrations result in slow reaction and incomplete curing with a resulting product of high moisture and free acid content.

Temperature is also an important process variable. Its effect interferes with acid concentration because the reaction is exothermic. Acid concentrations above 75% H_3PO_4 increase the rate of reaction, and an excessive amount of heat is liberated within a short time in the first stage of the reaction. Temperatures above 284° F. may decompose the monocalcium phosphate into unavailable pyrophosphate (*11*). Rapid rise in temperature of the freshly acidulated mass tends to drive off water, increasing the concentration of the acid. The increased concentration means further increase in reaction rate, and hence higher temperature of the mixture. Thus a balance is required between the temperature and the acid concentration such that optimum conditions are obtained in the first stage of the reaction. Various workers (*2–4*, *6*, *15*) have suggested limiting temperatures in the range of 150° to 302° F.

Acidulation ratio is less important as a process variable than as an economic factor. An acidulation ratio of 2.0 is the theoretical minimum based on stoichiometry. Generally, it is necessary to use an acidulation ratio greater than theoretical due to the loss of acid consumed by impurities and side reactions.