to reduce the total tonnage of calcium, magnesium, and sulfur applied in mixed fertilizers.

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FERTILIZER MATERIALS

Effect of Particle Size on the Granulation of Triple Superphosphate

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Granulation efficiency of triple superphosphate during ammoniation was best when the initial material had a low proportion (5 to 15%) of coarse (6–20 mesh), a high proportion (65 to 85%) of intermediate (20–65 mesh), and a relatively low proportion (10 to 20%) of fine (−65 mesh) particles. A higher proportion of coarse particles was detrimental to particle growth, and a higher proportion of fines made the material sensitive to slight variations in moisture content. A triple superphosphate with hard, discrete particles granulated more efficiently than one with soft, fragile particles and indicated greater response to changes in initial average particle size. Both the pattern of size distribution and the average size of raw materials are determining factors in granulation efficiency.

CONCERN ABOUT THE PARTICLE SIZE of raw materials used in fertilizer granulation processes is evident in recent technical literature (*1, 4, 10, 12, 14, 18, 20*). Standardization of the particle size of raw materials and the effects of particle size on granulation efficiency are subjects of current interest to the fertilizer industry (*1, 10, 12*). Particle-size requirements depend somewhat on the grade of fertilizer being granulated (*16–18*). Grades low in nitrogen and high in potash, such as 5–20–20 fertilizer, are reported to be difficult to granulate without the use of rather coarse potassium chloride; whereas high nitrogen grades, such as 10–10–10, appear to granulate well with finer potassium chloride (*10–12, 16, 18, 20*). There are indications that coarse superphosphate may be substituted to some extent for coarse potassium chloride as a granulation aid (*16, 18*).

As a consequence of industrial granulation experience, three size classes of potassium chloride have become available on the market (*20*)—granular, coarse, and regular. Granular potassium chloride, frequently used in processes for granulating low-nitrogen grades, is known to improve both the movement of material through rotary equipment and the yield of on-size product. However, it interferes with uniformity of nutrient distribution in the product (*6, 7, 11, 13*). Fine potassium chloride, used in the nonslurry process for granulating mixed fertilizers, often fails to be incorporated into the granule and frequently overloads the processing equipment with recycle material composed largely of potassium chloride.

Industrial experience with potassium chloride and experimental use of both potassium chloride and superphosphate in the granulation of mixed fertilizer indicate that each kind or type of solid ingredient may have a particle-size distribution pattern that is optimum for the most efficient granulation of the mixture.

The effects of variations in the initial particle-size distribution pattern and average size of two triple superphosphates on granulation efficiency are reported here.

Properties of Triple Superphosphate

Chemical and physical properties of triple superphosphates *A* and *B* are given in Table I. P_2O_5 and moisture were determined by official A.O.A.C. procedure (*2*). Free acid was determined by the acetone extraction method (*9*). Density measurements of three types were conducted on 10- to 20-mesh particles. True density represents the weight per unit volume of the solid and liquid phase components and was determined by helium displacement. Particle density represents the weight per unit volume of the solid and liquid phase components including voids exhibiting pore diameters of less than 100 microns, which is approximately the size of the openings of a 150-mesh sieve. Particle density was determined by mercury displacement. Bulk density represents the weight per unit volume of the solid and liquid components, including pore volumes and voids between particles, and was determined with the use of a glass cylinder, 5.2 cm. in diameter and 20.4 cm. high. The values for pore volume were calculated from the reciprocals of density measurements according to the formulas shown in Table I.

Particle hardness was determined by crushing-strength tests (*7*), and is represented by the average crushing strength of 100 6- to 8-mesh particles.

The triple superphosphates were quite

Table I. Chemical and Physical Properties of Triple Superphosphates

Properties	Triple Superphosphates A	Triple Superphosphates B
Chemical	Per Cent	
Total P_2O_5	49.26	50.07
Citrate-insoluble P_2O_5	0.34	0.04
Available P_2O_5	48.92	50.03
Water-soluble P_2O_5	42.25	41.35
Free acid (as H_3PO_4)	0.52	1.22
Free moisture	1.38	2.69
Physical	Grams/Cc.	
True density (T. D.)	2.188	2.297
Particle density (P. D.)	1.567	1.595
Bulk density (B. D.)	0.753	0.738
Volume of pores less than 100 microns (1/P. D. − 1/T. D.)	Cc./Gram 0.181	0.192
Volume of pores greater than 100 microns plus voids (1/B. D. − 1/P. D.)	0.690	0.728
Particle crushing strength	Lb. 5.3	2.3

Table II. Initial Particle Size Distribution Patterns

Pattern No.	Pattern Size levels	Particle Size Analyses[a]				
		Mesh				
		6–10	10–20	20–35	35–65	65–150
		Per Cent				
IV_{ax}	Coarsest	7	17	25	22	29
		Mesh				
		(6)–14	14–28	28–48	48–100	100–(270)
		Per Cent				
I_a	Coarse	14	20	21	28	17
II_a	Coarse	12	26	28	29	5
III_a	Coarse	6	17	46	28	3
IV_a	Coarse	7	15	20	32	26
		Mesh				
		10–20	20–35	35–65	65–150	150–270
		Per Cent				
I_b	Medium	18	20	20	21	21
II_b	Medium	13	27	37	18	5
III_b	Medium	5	16	59	18	2
IV_b	Medium	7	17	25	22	29
		Mesh				
		(10)–28	28–48	48–100	100–200	200–(270)
		Per Cent				
I_c	Fine[b]	15	19	31	24	11
II_c	Fine[b]	11	22	44	19	4
III_c	Fine[b]	6	13	67	12	2
IV_c	Fine[b]	8	14	36	30	12

[a] With the exception of terminal sieves (shown in parenthesis) for coarse and fine size levels, patterns are classified in terms of either of the two sets of sieves resulting from the combination of alternate sieves of the Tyler $\sqrt{2}$ Screen Scale Series.

[b] The fine patterns were formulated with supplemental proportions of 150–270 mesh corresponding to the proportions finer than 200 mesh in the medium patterns, because no material finer than 270 mesh was available.

Table III. Accuracy of Average Size Development within Pattern

Pattern Number	Average Size, Mm. a, Coarse	Average Size, Mm. b, Medium	Average Size, Mm. c, Fine	Size Ratio[a] a/b	Size Ratio[a] b/c
		Triple Superphosphate *A*			
I	0.370	0.270	0.181	1.37	1.49
II	0.449	0.334	0.218	1.34	1.53
III	0.342	0.259	0.181	1.32	1.43
IV[b]	0.258	0.194	0.138	1.33	1.41
		Triple Superphosphate *B*			
I	0.356	0.251	0.176	1.42	1.43

[a] Theoretical size ratio is 1.41 ($\sqrt{2}$).

[b] Supplementary coarsest version, IV_{ax} of this pattern had an average size of 0.399 mm. 0.399/0.194 = 2.06; theoretical = 2.00.

different in particle structure. Superphosphate *B* exhibited a crushing strength only 43% of that of *A*, and was more porous. Visual observations of the magnified particles verified the prevalence of large pits on the surfaces of superphosphate *B*. Such pits are considered significant to the character of the particle but are not readily measurable (*5*).

Particle Size Distribution Patterns

Each triple superphosphate (*A* and *B*) was sieved to obtain the following mesh size classes (Tyler): 6–10, 10–20, 20–35, 35–65, 65–150, and 150–270. Experimental patterns of particle size distribution were prepared from these stocks of size classes.

A standard pattern, I_b, of triple superphosphate *A*, characterized by hard, discrete particles, had approximately equivalent proportions in each of the five size classes in the 10- to 270-mesh range. Pattern II_b had increased proportions of medium-coarse (20–65 mesh) particles, Pattern III_b had increased proportions of medium (35–65 mesh) particles, and Pattern IV_b had increased proportions of fine (−65 mesh) particles (Table II). Each formulation was analyzed by means of a second, equivalent (Table II) set of sieves, 14, 28, 48, 100, and 200 mesh. The proportions resulting were used in conjunction with the six stock sizes in the 6- to 270-mesh range to replicate the initial patterns at a higher level of size, Patterns I_a-IV_a, and in conjunction with the five stock sizes in the 10- to 270-mesh range, to replicate the patterns at a lower level of size, Patterns I_c-IV_c (Table II). Accordingly, coarse, medium, and fine versions of each pattern were prepared which represented average particle sizes in the ratio of $2:\sqrt{2}:1$, respectively. Pattern IV_{ax}, representing an average particle size twice that of IV_b, was prepared because it eliminated −150-mesh material.

Triple superphosphate *B*, with soft, porous particles was prepared in three particle-size distributions of Pattern I.

Determination of Average Size

Particle-size distribution curves were obtained for each size-pattern by a procedure (*19*) in which the cumulative weight per cent retention on each sieve of the $\sqrt{2}$ series is plotted as a function of the sieve opening. This procedure is illustrated in Figure 1. The average sizes were taken to be the sieve opening, in millimeters, retaining 50% of the weight of the respective distributions, and may be ascertained by noting the points of intersection of the particle-size distribution curves with the 50% re-

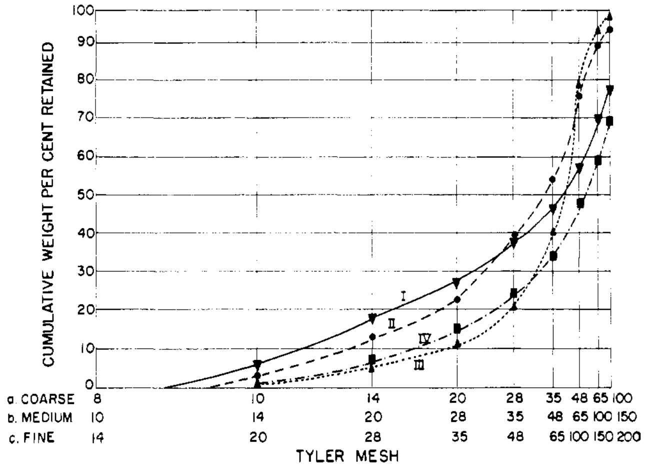

Figure 1. Initial particle size distribution patterns

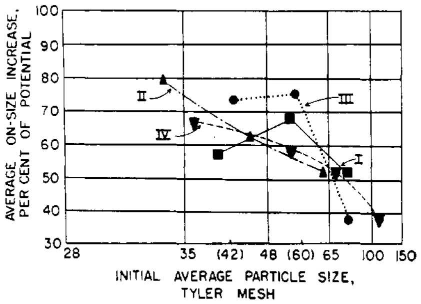

Figure 2. Efficiencies of on-size production

Table IV. Size Conformity between Patterns of Triple Superphosphate *A*

Ratios of Pattern Numbers	*a*, Coarse patterns[a]	*b*, Medium patterns[b]	*c*, Fine patterns[c]	Average
	Ratios of Average Size			
I/II	0.82	0.81	0.83	0.82
I/III	1.08	1.04	1.00	1.04
I/IV	1.43	1.39	1.31	1.38

[a] I_a divided by II_a, III_a, and IV_a, respectively.

[b] I_b divided by II_b, III_b, and IV_b, respectively.

[c] I_c divided by II_c, III_c, and IV_c, respectively.

tention line. An average size so determined represents the geometric mean diameter, a concept which has been effectively applied in particle-size analysis of other substances, such as silica, granite, calcite (*8*), and clay (*15*).

The initial average sizes of the particle-size distribution patterns for each triple superphosphate material are given in Table III. The size-ratios are a measure of the accuracy of the size differences within the patterns. The deviation from the theoretical size-ratio probably resulted from some drying and breakdown in particle size which occurred during the study. Conformity of size-ratios between patterns for shifts in average size from medium to coarse and fine, respectively, appears satisfactory (Table IV).

Granulation Experiments

Six-hundred gram batches of each size were wetted, mixed, and charged to a preheated rotary drum for ammoniation (4 pounds per unit of P_2O_5) and granulation at 100° C. Subsequent batches were wetted until the moisture content just exceeded that required for maximal production of on-size (6–20 mesh) granules as evidenced by excessive production of over-size (+6 mesh) granules. Each batch was air-dried overnight and sieved, and the average size was determined.

Granulation efficiencies were evaluated in terms of: the sensitivity of granulation as reflected by variation in the range of moisture content for which acceptable increases in the proportion of on-size granules were obtainable; the average increase in the proportion of on-size; and the relative granule growth for an arbitrarily assigned optimal range of moisture content as shown in Table V.

The optimal moisture content for each size-pattern was taken as the amount of moisture resulting in maximum yields of on-size material, provided not more than 10% over-size was present. When the optimum moisture content was exceeded only slightly, over-agglomeration occurred extremely rapidly. The tendency to form over-size may be ascertained indirectly by the sensitivity of granulation to changes in moisture range (Table V)—the wider the moisture range the less the tendency to form over-size.

On-size production was considered acceptable if the increase in on-size was more than 60% of the potential increase. On-size production was expressed as per cent of the potential increase. This avoids bias in favor of patterns representing materials containing a high proportion of initial particles of the same size as that desired in the granular product. The various patterns represent materials initially containing from 2 to 27% of 6- to 20-mesh particles.

Relative granule growth is a measure of the degree of agglomeration of initial particles, and as such, is considered a measure of the probability of approachment to uniformity of nutrient distribution among product granules. The assumption is made, therefore, that the greater the relative growth the greater the probability that particles of the various nutrient materials will be uniformly distributed within the resultant granules. A satisfactory degree of granule growth would appear to be especially important in the processing of mixtures where one or more of the constituent materials has a tendency to resist agglomeration.

Experimental Results

Granulation efficiencies varied widely within type of triple superphosphate, owing to changes in pattern of particle size distribution and average size, and between types, owing to differences in particle structure (Table V).

For two fine patterns, III_c and IV_c, of the hard, discrete particles of type *A*, no moisture range produced acceptable increases in on-size product. The other two fine patterns, I_c and II_c, exhibited very low tolerances of moisture variation for which on-size increases were acceptable, 99 to 100% and 97 to 100% of optimal moisture, respectively. For patterns in the medium size range, however, granulation was at acceptable levels of on-size for a moisture range as wide as 85 to 100% of the optimal moisture content (Table V, pattern III_b). The optimal initial average size for best performance in this regard varied with the individual patterns; however, each pattern appeared to perform satisfactorily for a size range, generally, between 0.300 mm. (about 48 mesh) and 0.250 mm. (about 60 mesh). To compare the efficiencies of on-size production for individual patterns, the average increases in on-size (column 6) were plotted, in Figure 2, as a function of the corresponding initial average sizes (column 2). These data indicate that for initial average sizes below 0.208 mm. (65 mesh), on-size production was unsatisfactory,—i.e., less than 60% of the potential, for each pattern. For the rather wide medium range of average size from 0.351 mm. (42 mesh) to 0.246 mm. (60 mesh), Pattern III was superior to the others, with on-size production from 73 to 76% of the potential. Pattern III exhibits a normal distribution, in that it was formulated with the greatest

Table V. Granulation Efficiencies of Triple Superphosphates

Initial Particle Size Distribution Pattern: No.	Initial Particle Size Distribution Pattern: Av. size, mm.	Optimal Moisture Content, %	Range of Moisture Content for Acceptable[a] Increases in On-Size, % of Optimal	Granulation at 90–100% of Optimal Moisture Content: Increase in On-size, % of Potential: Range	Av.	Final average size, mm.	Relative granule growth, $\frac{V_f - V_i}{V_i}$ [b]
			Triple Superphosphate *B* (Soft Porous Particles)				
I_a	0.356	16.8	94–100	51–64	55	1.163	34
$_b$	0.251	17.7	97–100	43–61	53	1.069	76
	0.176	15.4	None	38–48	42	0.887	126
			Triple Superphosphate *A* (Hard Discrete Particles)				
I_a	0.370	22.3	96–100	49–65	57	1.199	33
$_b$	0.270	22.1	90–100	60–74	68	1.210	89
$_c$	0.181	21.1	99–100	41–63	52	0.945	142
II_a	0.449	23.6	80–100	71–91	80	1.298	23
$_b$	0.334	25.4	93–100	49–71	63	1.045	30
$_c$	0.218	21.1	97–100	37–73	53	0.935	78
III_a	0.342	24.1	84–100	67–80	74	1.139	36
$_b$	0.259	25.3	85–100	69–82	76	1.316	130
$_c$	0.181	21.1	None	29–47	38	0.755	72
IV_{ax}	0.399	22.8	92–100	55–78	67	1.024	16
$_a$	0.258	22.3	97–100	47–76	58	1.094	75
$_b$	0.194	23.6	98–100	40–73	53	0.976	126
$_c$	0.138	20.2	None	27–55	38	0.758	167

[a] Increase in proportion of on-size (6–20 mesh) was more than 60% of the potential increase. The potential increase was the percentage finer than 20 mesh initially.

[b] Volume of the final average particle minus the volume of the initial average particle divided by the latter. Volumes were calculated on the assumption of sphericity of the respective average particles. Values from columns 2 and 7 were used.

weight proportion of particles in the middle (35–65 mesh) of the size range, and with the weight proportions tapering off on the coarser (+35 mesh) and finer (−65 mesh) portions of the size range (Table II). Pattern I, formulated with approximately equivalent amounts in each size class (Table II) was next to Pattern III in performance for the medium range of average size (Figure 2). For average sizes increasing above 0.351 mm., only Patterns II and IV, having larger proportions of medium-coarse (20–65) and fine (−65) particles, respectively (Table II), indicated greater efficiency of on-size production, and the increase in the case of Pattern IV was only slight. Pattern II, in a very coarse range of average size above 0.417 mm. (35 mesh), would appear to be an excellent pattern (Figure 2) in granulation situations where maximum on-size production is desired and a low degree of relative granule growth may be tolerated without adversely affecting the uniformity of nutrient distribution in the granular product. The patterns at the respective low levels of average size exhibited high relative granule growth owing to the high proportions, 45 to 66%, of particles finer than 65 mesh, but low production of on-size owing to the excessive plasticity produced by small increments of moisture, resulting in over-agglomeration. The pattern initially having a normal distribution of particle sizes, III_b, best fulfilled requirements of maximum production of on-size granules and a high degree of granule growth, with average increases in on-size of 76% of the potential and a relative granule growth ratio of 130. In general form, this best-performing pattern may be represented as follows: 5 to 15% of 6–20 mesh, 65 to 85% of 20–65 mesh, and 10 to 20% of −65 mesh. A higher proportion of coarse particles was detrimental to particle growth and a higher proportion of fine particles made the material sensitive to slight variations in moisture content.

The hard, discrete particles of the *A* material absorbed moisture readily and remained intact; whereas the soft, porous particles of the *B* material resisted moisture penetration and appeared to collapse. For size-pattern I_b, superphosphate *B* evidenced acceptable granulation for a range of moisture content of only 97 to 100% of the optimal as compared with 90% to 100% for superphosphate *A* (Table V). Granulation of the *A* material appeared to be more readily controlled, under industrial conditions, than granulation of the *B* material. The differences in wetting behavior and granulation performance are attributed to the structural differences between particles of the two materials. Hardness, density, and porosity of particles are reported to considerably influence granulation of triple superphosphate alone or in admixture with other fertilizer ingredients (*3–5*, *14*).

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