

SUPERPHOSPHATE AMMONIATION

Physical Factors Influencing Ammonia Absorption by Superphosphates

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The trend toward production of higher analysis fertilizers and the comparatively low cost of neutralizing ammonia have led the mixed-fertilizer producer to attempt higher and higher rates of ammoniation. This has created problems in ammoniation technique related to the maintenance of optimum conditions for ammoniation and the influence of physical properties of superphosphates on ammonia absorption. The present study evaluates factors affecting the amount of ammonia that superphosphates will absorb. Absorption efficiency increases from 70 to 96% as the initial moisture content of the superphosphate is increased from 1 to 7%. The efficiency increases from 84 to 100% as the mean particle size of the superphosphate is decreased from 2.5 mm. (6- to 10-mesh) to 0.1 mm. (minus 80-mesh). Denseness and hardness of superphosphate particles, low reaction temperatures, and short reaction periods are factors contributing to poor ammonia absorption. The data should serve as a guide in the manufacture of mixed fertilizers containing high amounts of neutralizing ammonia.

INCORPORATION OF NEUTRALIZING AMMONIA in superphosphate by chemical combination has steadily increased since about 1935, when anhydrous ammonia and commercial ammoniating solutions had become well established as economical sources of fertilizer nitrogen. U. S. consumption data for the fiscal year 1949 show that 170,000 tons of neutralizing ammonia (76) and 1,368,200 tons of available phosphorus pentoxide (22) in superphosphate were used in mixed fertilizers, or an average of 2.5 pounds of ammonia for each 20-pound unit of phosphorus pentoxide.

Under favorable conditions, the maximum amounts of neutralizing ammonia which react with superphosphates are 9.6 pounds per 20 pound unit of phosphorus pentoxide in ordinary superphosphate and 6.4 pounds per unit of phosphorus pentoxide in triple superphosphate. These values are in agreement with equations for the ammoniation reaction proposed by Keenen (14), Hardesty and Ross (6), and White *et al.* (23). In the early days manufacturers hesitated to ammoniate beyond 3 pounds of ammonia per unit of phosphorus pentoxide because of possible losses of nitrogen and available phosphorus dur-

ing ammoniation and subsequent storage of the ammoniated product. More recently, increasing demands for high-analysis fertilizers and the comparatively low cost of neutralizing ammonia have led the mixed-fertilizer producer to attempt higher and higher rates of ammoniation (5).

The importance of ammoniation to the fertilizer industry and to the farmer is reflected by numerous publications on the mechanism of chemical reactions between ammonia and superphosphates (7, 3, 6, 14, 23), the properties of citrate-insoluble residues from ammoniated products (4, 9, 10, 12, 13, 15, 27), and the agronomic efficiency of ammoniated products as compared with other phosphate fertilizers (8, 18-20). However, there is a dearth of published information on ammoniation technique, optimum conditions for ammoniation, and the influence of physical properties of raw materials on ammonia absorption.

The present study deals chiefly with some physical factors that may influence the amount of ammonia absorbed by superphosphate. Such factors include density, particle size, structure, and free moisture content of the superphosphates; temperature of ammoniation; period of

contact with ammonia; and concentration of phosphorus pentoxide in the superphosphate or fertilizer mixture being ammoniated.

Equipment and Procedure

The laboratory investigations were carried out with the equipment shown in Figure 1. Anhydrous ammonia stored in a 2-gallon stainless steel tank was drawn into a calibrated steel pipe, equipped with sight glass, from which a measured amount was dispensed into the ammoniator. The ammoniator, a steel drum 7 inches wide and 14 inches in diameter, with three equally spaced 1-inch cross flights, rotated at 30 r.p.m. Temperature changes were measured by a fixed iron-constantan thermocouple connected to a temperature recorder. A batch consisting of 600 grams of material was wetted to the desired moisture content, mixed, and stored for 24 hours to permit equilibration of the moisture. Rate of injection of ammonia was manually controlled with the use of a stop-watch. Injection over a 2-minute period was followed by an additional 2 minutes of mixing to give a total reaction period of 4 minutes

Table I. Properties of Superphosphates

Physical Properties			Chemical Analyses ^a , %				
Screen Analysis		Apparent density, lb./cu. ft.	Phosphoric Oxide (P ₂ O ₅)				
Tyler mesh range	Fraction present, %		Total	Citrate insoluble	Available	Water soluble	Free acid (as H ₃ PO ₄)
Ordinary Superphosphate							
Plus 4	0.0	...	24.49	0.57	23.92	20.48	0.43
4 to 6	1.1	45.6	21.42	0.11	21.31	18.03	0.77
6 to 10	9.4	43.4	20.60	0.27	20.33	17.62	1.72
10 to 20	11.7	45.5	20.98	0.07	20.91	18.43	2.29
20 to 40	11.8	43.9	22.15	0.06	22.09	19.25	1.66
40 to 80	30.0	43.2	18.99	0.06	18.93	15.08	2.03
Minus 80	36.0	40.1	20.65	0.10	20.55	18.35	1.78 ^c
Composite	100.0	42.5 ^b					
Triple Superphosphate, Type A							
Plus 4	0.0	...	36.92	0.09	36.83	31.00	0.12
4 to 6	0.7	39.2	46.23	0.03	46.20	40.01	0.14
6 to 10	6.1	45.9	48.63	0.01	48.62	42.47	0.14
10 to 20	16.8	46.0	48.53	0.00	48.53	42.17	0.14
20 to 40	23.3	48.7	48.75	0.02	48.73	42.31	0.13
40 to 80	28.9	50.9	41.06	0.01	41.05	33.12	0.14
Minus 80	24.2	48.0	45.86	0.01	45.85	39.85	0.14 ^c
Composite	100.0	49.1 ^b					
Triple Superphosphate, Type B							
Plus 4	0.4	...	48.52	0.69	47.83	46.27	2.41
4 to 6	3.9	49.1	48.72	0.47	48.25	46.24	2.70
6 to 10	11.3	51.2	48.61	0.53	48.08	46.32	2.79
10 to 20	20.8	54.3	48.41	0.90	47.51	44.52	2.77
20 to 40	25.7	57.0	49.17	0.66	48.51	44.19	2.42
40 to 80	24.0	60.8	48.09	0.07	48.02	43.53	3.02
Minus 80	13.9	60.4	48.64	0.59	48.05	44.97	2.69 ^c
Composite	100.0	56.8 ^b					

^a Moisture-free basis. The original moisture contents of the three superphosphates were, respectively, 1.76, 0.79, and 3.87%.
^b Crushing strength values for particles in ordinary, Type A, and Type B superphosphates were 25, 78, and 122 lb./sq. inch, respectively.
^c Fluorine contents of the ordinary, Type A, and Type B superphosphates were 1.74, 1.82, and 2.18%, respectively.

except where otherwise noted. This is considered consistent with the 3-minute cycle prevalent in industry on larger batches of material.

After discharge, the batch was exposed to the air for 5 minutes to permit escape of unreacted ammonia before being stored in Mason jars. Analytical samples were obtained after 24 hours. Standard AOAC methods of analysis (2) were used for free moisture (air-flow procedure), nitrogen, and phosphorus pentoxide determinations. Free acid was determined by the acetone method described by Hill and Beeson (77).

Table II. Effect of Ammoniation Rate on Ammonia Absorption Efficiency of Superphosphates

Ammonia Added lb. NH ₃ / 20 lb. P ₂ O ₅	% of Added Ammonia Absorbed by Superphosphates	
	Ordinary ^a	Triple ^b
2	...	99.2
3	99.8	94.4
4	...	92.6
5	96.0	82.7
6	93.5	...
7	83.7	...

^a Initial moisture, 6.5%.
^b Type A, initial moisture, 5.2%.

Fluorine was determined by the method described by Reynolds and Hill (77).

Description of Superphosphates

The superphosphates selected for the laboratory studies were commercial products prepared by acidulation of Florida land pebble rock phosphates. The ordinary and the Type A triple superphosphates (Table I) were composed chiefly of soft, porous particles, while Type B triple superphosphate was a more granular product composed chiefly of hard, dense particles. Compositions of the composite samples were calculated from the analyses of their fractions. Crushing strength values were determined by the method described by Hardesty and Ross (7).

Ammoniation Rate

The term "ammoniation rate" is widely used in the fertilizer industry to express the pounds of neutralizing ammonia added per unit, or 20 pounds, of phosphorus pentoxide in the superphosphate. The term "degree of ammoniation" is often used synonymously with "rate," but in this paper the degree of ammoniation refers to the pounds of ammonia absorbed per 20 pounds of phosphorus pentoxide. Absorption effi-

ciency refers to the amount of ammonia absorbed in terms of per cent of that added.

Representative samples of superphosphates were ammoniated under controlled conditions to show the effect of various ammoniation rates on absorption efficiency. Table II shows that an increase in the ammoniation rate decreases the absorption efficiency. It also indicates that comparable efficiencies should be obtained with ordinary superphosphate ammoniated at 6 pounds per unit of phosphorus pentoxide and triple superphosphate ammoniated at 4 pounds per unit. Accordingly, the absorption efficiencies of ordinary superphosphate and the Type A triple superphosphate, ammoniated to 6 and 4 pounds per unit of phosphorus pentoxide, respectively, were compared over a varying range of moisture content. Absorption values reported in this paper are characteristic only of the rotary mixer previously described.

Initial Moisture Content

The curve in Figure 2 shows that absorption efficiency increases from about 70% to 96% as the initial moisture content of the superphosphates is increased from 1 to 7%. Figure 2 also shows that in the presence of about 6%

free moisture, ordinary superphosphate can be ammoniated to 6 pounds of ammonia per unit of phosphorus pentoxide, and Type A triple superphosphate to about 4 pounds per unit with approximately 95% absorption efficiency in each case. In all subsequent series of tests moisture contents were maintained at or about 6%. In commercial practice the advantages of high moisture content for ammoniation are weighed against the disadvantages of additional processing—i.e., drying or addition of conditioners, which would be required to achieve the same degree of physical condition in the final product.

Particle Size

The effect of particle size of superphosphate on ammonia absorption was determined by fractionating ordinary superphosphate (Table I) into convenient mesh sizes and wetting and ammoniating each fraction separately. Each batch was ammoniated to 6 pounds of free ammonia per unit of available phosphorus pentoxide. Curve 1, Figure 3, shows that only the minus 80-mesh fraction of ordinary superphosphate absorbed all the ammonia added. The other fractions in the order of increasing mesh size showed a decreasing capacity for absorption, the coarsest, 5- to 10-mesh fraction, absorbing only 5 pounds per unit of phosphorus pentoxide. Curve 2 shows that ammonia is preferentially absorbed by the smaller particles when all size fractions are present during ammoniation. This curve was obtained by ammoniating a composite of 4-6, 6-10, 10-20, 20-40, 40-80, and minus 80-mesh fractions, each containing the same amount of available phosphorus pentoxide. After ammoniation with 6 pounds of free

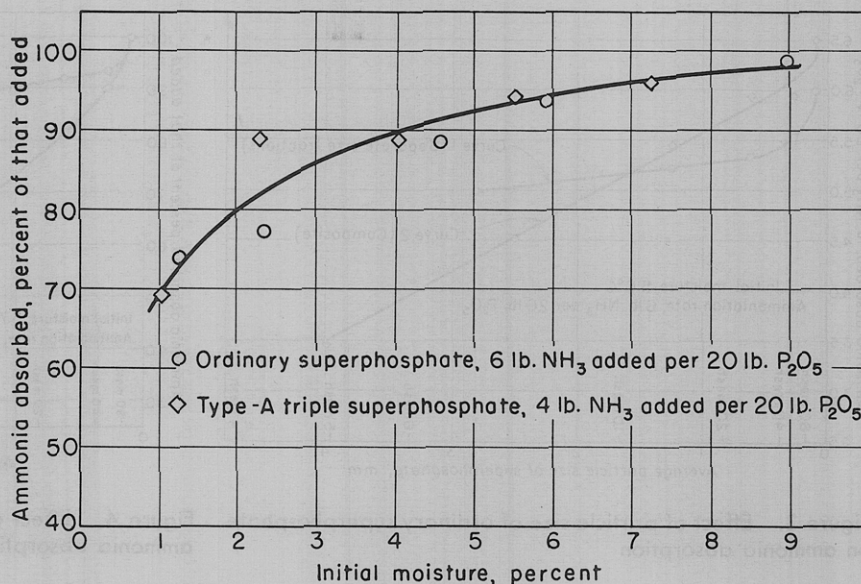


Figure 2. Effect of initial moisture content of superphosphates on ammonia absorption efficiency

ammonia per unit of phosphorus pentoxide, the sample was refractionated and each fraction analyzed separately. Although the ammoniation rate was 6 pounds of ammonia per unit of phosphorus pentoxide, the actual degree of ammoniation was 6.5 pounds per unit for the minus 80-mesh particles and 3.4 pounds per unit for the 4- to 6-mesh particles.

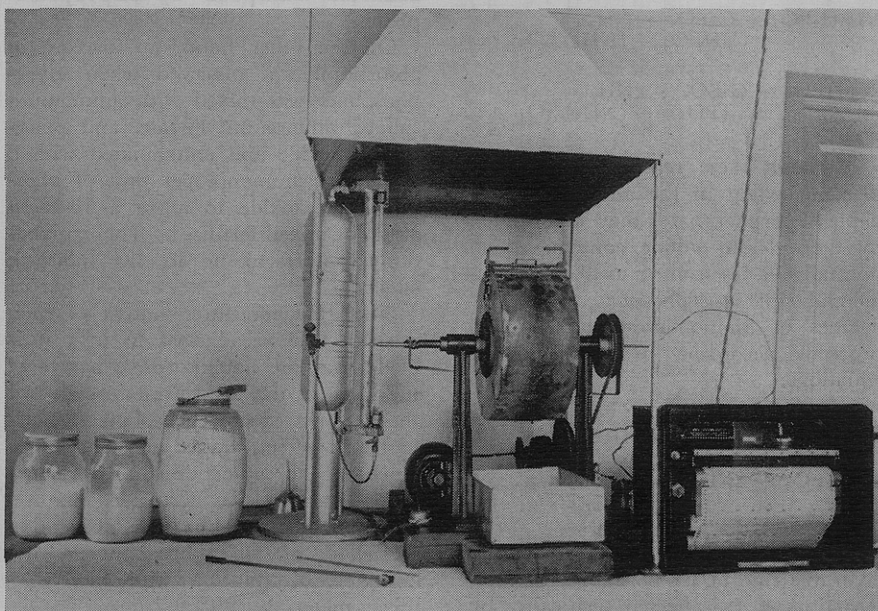
Density and Hardness

Figure 4 shows the variation in absorption efficiency with particle size for the two different types of triple superphosphate described earlier. These superphosphates of different apparent density and hardness were fractionated as shown in Table I and each fraction

treated with 4 pounds of ammonia per unit of phosphorus pentoxide.

By comparison with curve 1 of Figure 3 it may be seen that absorption characteristics of Type A triple superphosphate parallel those of the ordinary superphosphate. Type B triple superphosphate, however, offers much greater resistance to ammoniation. Although the minus 80-mesh fractions of both triple superphosphates attain 100% absorption, the 6- to 10-mesh fraction of Type B absorbs only 49% as compared to 85% for the corresponding size fraction of Type A. No differences are observed in chemical composition of these two superphosphates (Table I) that would account for differences in absorption capacity, except that Type B has higher free acid and available and water-soluble phosphorus pentoxide contents than Type A; and on this consideration alone should have greater ammonia-absorbing capacity. The fact that its absorbing capacity is low points to differences in the physical properties of the two superphosphates. Type B has greater apparent density and granule hardness, and a more encrusted particle than Type A (Table I). Apparently, many ammoniation difficulties encountered in commercial practice are attributable to a high proportion of dense, hard encrusted granules in the superphosphate.

Figure 1. Ammoniation equipment



Reaction Period

Different batches of ordinary superphosphate containing 7% moisture were ammoniated at rates of 4 and 7 pounds of ammonia per unit of phosphorus pentoxide for reaction periods varying from 1 to 8 minutes. Figure 5 shows that an increase in the reaction period from

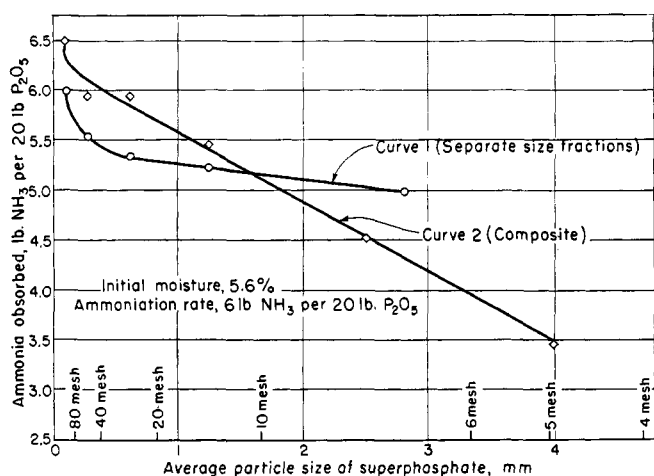


Figure 3. Effect of particle size of ordinary superphosphate on ammonia absorption

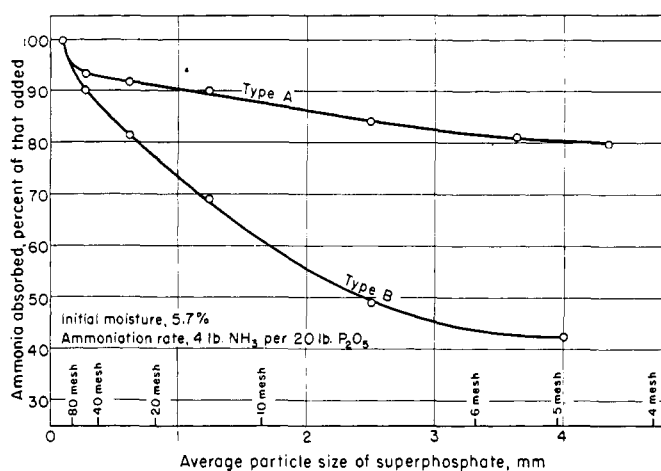


Figure 4. Effect of particle size of triple superphosphate on ammonia absorption efficiency

1 to 8 minutes at the 4-pound rate of ammoniation increased the absorption efficiency from 97 to 100% and, at the 7-pound rate, from 72 to 88%. The higher absorption efficiencies were substantially obtained in 1.5 minutes at the 4-pound ammoniation rate and in 4 minutes at the 7-pound rate.

Reaction Temperature

In the foregoing tests no effort was made to control the final reaction temperatures and the maximum temperature attained in each case was that due to the heat of reaction. Room temperature varied from 68° to 82° F. and final reaction temperatures from 113° to 163° F. depending on the type of superphosphate, amount of moisture present, and degree of ammoniation.

Table III. Effect of Reaction Temperature on Ammonia Absorption Efficiency of Superphosphates

Superphosphate and Ammoniation Rate	Controlled Max. Temp. of Reaction, ° F.	% of Added Ammonia Absorbed
Ordinary, ^a	122	83.5
7 lb. NH ₃ /20 lb. P ₂ O ₅	138	83.7
	190	83.4
Triple, ^b	149	79.1
5 lb. NH ₃ /20 lb. P ₂ O ₅	163	83.0
	216	89.6

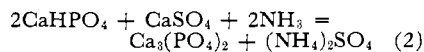
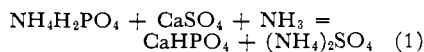
^a Initial moisture, 6.4%.

Type A, initial moisture, 5.2%.

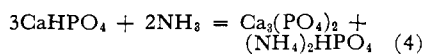
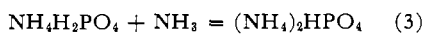
To determine the effect of temperature on absorption efficiencies, ordinary superphosphate was treated with 7 pounds of ammonia per unit of phosphorus pentoxide, and Type A triple superphosphate was treated with 5 pounds at several different temperatures which were obtained by the application

of external heat to the ammoniation drum. These exceptionally high rates of ammoniation were used in order to emphasize any increase in absorption that might be attributed to high temperature. Table III shows that raising the maximum reaction temperature from 122° to 190° F. caused no change in ammonia absorption by ordinary superphosphate. In the case of triple superphosphate raising the reaction temperature from 149° to 216° F. increased the absorption efficiency from 79.1 to 89.6%.

The primary products in the ammoniation of the monocalcium phosphate in both ordinary and triple superphosphates are dicalcium phosphate and monoammonium phosphate (6, 14). In the ammoniation of ordinary superphosphate beyond 2.3%, these primary products react with calcium sulfate to form more basic phosphate and ammonium sulfate according to the equations:



Although these reactions also occur to some extent in the ammoniation of triple superphosphate, they are limited by the calcium sulfate content, which normally varies from 1 to 4% in commercial triple superphosphate. At higher degrees of ammoniation the reaction proceeds according to the following equations:



Although Reaction 3 occurs at a favorable rate under normal conditions, Reaction 4 is affected critically by

temperature. White *et al.* (23), showed that dicalcium phosphate containing 5% moisture absorbed at 140° F. about 11% and at 160° F. about 41% of the ammonia absorbed at 212° F. These data indicate the necessity for taking into account the ammoniation temperature if high degrees of ammoniation are to be obtained, especially in the case of triple superphosphate.

Diluents and Soluble Salts

Earlier workers (8, 14, 15, 27) have shown that loss of phosphate availability through reversion is significantly decreased when superphosphate is ammoniated in the presence of other materials. To determine the effect of such dilution of superphosphate on the absorption of ammonia, ordinary superphosphate was mixed with dry sand and treated with 6 pounds of ammonia per unit of phosphorus pentoxide. The dilution substantially increased the ammonia absorption by superphosphate.

On the other hand, no increase in absorption was observed when superphosphate was mixed with ammonium sulfate, ammonium nitrate, and potassium chloride and ammoniated with 6 pounds of ammonia per unit of phosphorus pentoxide to make a 10-10-10 grade of mixed fertilizer. The explanation appears to lie in the moisture effects.

Since the moisture content of each mixture was preadjusted to 6%, it is likely that the nonabsorptive nature of the sand in the former mixture caused the interspersed superphosphate particles to be, in effect, wetter and thus more amenable to absorption of ammonia than those in the fertilizer mixture. The tests indicate that the absorption efficiency of superphosphate is not inhibited by dilution with these fertilizer materials.

Phosphate Solubilities

Factors which affect phosphate reversion are the degree of ammoniation; the chemical composition of the superphosphate, such as free acid and fluorine contents; presence of foreign matter; and storage conditions, such as length of storage and the temperature and moisture content of the stored product (4, 8, 9, 12, 13, 21).

The ammoniated ordinary and Type A triple superphosphates described in Table II were stored at 86° F. and analyzed after 30 days. They contained approximately 5% moisture during storage. Results of analyses of the original superphosphates are given in Table I. Figure 6 shows the changes in water-soluble and citrate-insoluble forms of phosphorus pentoxide that occur with increasing amounts of ammonia absorbed. The water-soluble and citrate-insoluble phosphorus pentoxide contents of ammoniated ordinary superphosphate are straight-line functions of the amount of ammonia absorbed and are 33 and 6%, respectively, of the total phosphorus pentoxide in superphosphate containing 6 pounds of ammonia per unit of phosphorus pentoxide. With triple superphosphate, the absorption of ammonia up to 2 pounds per unit of phosphorus pentoxide was accompanied by rapid changes in water-soluble and citrate-insoluble forms of phosphorus pentoxide, but further absorption up to 4.3 pounds of ammonia per unit of phosphorus pentoxide had little or no additional effect. At the higher ammoniation rates 59% of the total phosphorus pentoxide was water-soluble and 10% was citrate-insoluble.

The difference in the water-soluble

phosphorus pentoxide curves is characteristic for the ammoniation of ordinary and triple superphosphates and is readily explained by the equations given here. Calcium sulfate takes part in the reactions involved in the ammoniation of ordinary superphosphate at high rates (Equations 1 and 2) to decrease the content of water-soluble phosphorus pentoxide. In the ammoniation of triple superphosphate (Equations 3 and 4) water-soluble phosphorus pentoxide is retained in the form of ammonium phosphates irrespective of the degree of ammoniation, owing to the virtual absence of calcium sulfate.

Summary

Ordinary and triple superphosphates were ammoniated under controlled conditions in a closed rotary cylinder to determine the effects of some physical factors on ammonia absorption. Absorption efficiencies were materially increased by increase in moisture content and decrease in particle size of the superphosphate. An increase in the moisture content from 1 to 7% increased the absorption efficiency of ordinary superphosphate treated with 6 pounds of ammonia per unit of phosphorus pentoxide from 70 to 96%. A decrease in mean particle size of the superphosphate from 2.5 mm. (6- to 10-mesh) to 0.1 mm. (minus 80-mesh) increased the absorption efficiency from 84 to 100%. Denseness and hardness of superphosphate particles were factors contributing to poor ammonia absorption. The absorption efficiency of a hard, dense triple superphosphate decreased from 100 to 49% as the particle size was increased from minus 80- to 4-6-mesh.

Figure 5. Effect of reaction period on ammonia absorption efficiency

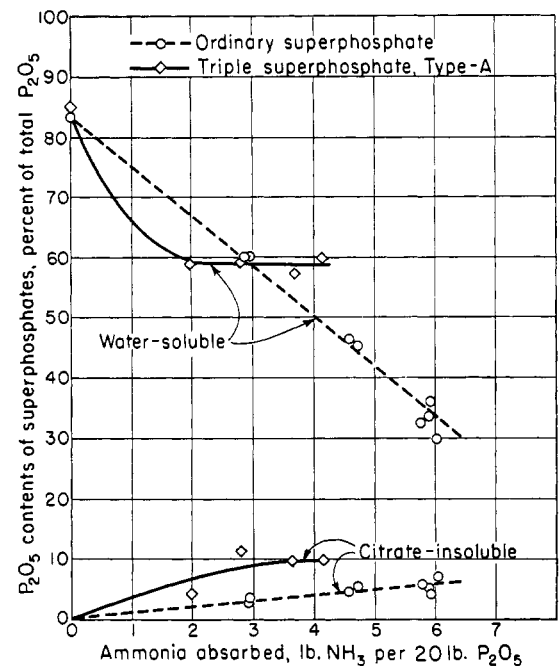
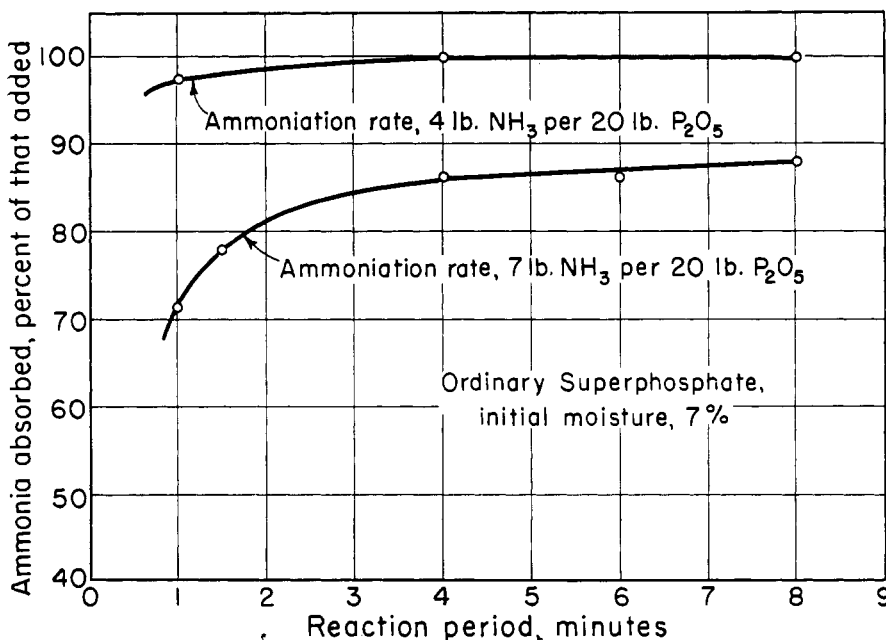


Figure 6. Effect of degree of ammoniation on solubilities of P₂O₅ in superphosphates

That of a softer, more porous superphosphate decreased only from 100 to 85% for same change in particle size.

Prolonging the reaction period from 1 to 4 minutes made very little difference in the absorption efficiency of ordinary superphosphate treated with 4 pounds of ammonia per unit of phosphorus pentoxide, but increased absorption from 72 to 88% for the same superphosphate at an ammoniation rate of 7 pounds per unit.

Raising the reaction temperature from 122° to 190° F. in the treatment of ordinary superphosphate with 7 pounds of ammonia per unit of phosphorus pentoxide gave no significant difference in absorption. However, in the case of triple superphosphate treated with 5 pounds of ammonia per unit of phosphorus pentoxide, raising the temperature from 149° to 216° F. increased the absorption from 79 to 90%. Dilution of ordinary superphosphate with other fertilizer materials to form a 10-10-10 mixed fertilizer did not alter its absorption capacity. Analysis of ammoniated ordinary superphosphate stored at 86° F. for 30 days showed that decrease in water-soluble and increase in citrate-insoluble forms of phosphorus pentoxide attributable to ammoniation were straight-line functions of the amount of ammonia absorbed. At an ammonia absorption of about 6 pounds per unit of phosphorus pentoxide, the water-soluble phosphorus pentoxide content was 33% and the citrate-insoluble phosphorus pentoxide content was 6%. With triple superphosphate, the water-soluble and citrate-insoluble phosphorus pentoxide

contents leveled off to values of 58 and 10%, respectively, at an absorption of about 2 pounds of ammonia per unit of phosphorus pentoxide.

In general, the physical conditions found to be most conducive to efficient ammonia absorption by superphosphates were low ammoniation rates, high temperature (for triple superphosphate), prolonged reaction periods, and such attributes of the superphosphate as fineness, softness, and high moisture content.

Acknowledgment

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Economical Waste Disposal for Pear Canneries; Color of Alkaline Sugar Solutions Studied

SUGAR RECOVERY

Recovery of Sugars from Pear-Canning Waste

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A process for the recovery of sugars and other soluble solids from peelings, cores, and trimmings wasted in canning pears involves conversion of the milled waste into a calcium pectate gel from which a clear, pectin-free juice is easily pressed, removal of excess calcium by means of an ion-exchange resin, and removal of color by precipitation and by adsorption with activated carbon. The purified juice, when used as a sirup base for canning pears, replaces about one third of the refined sugar now used in canning this fruit. The pomace, which amounts to about 25% of the waste, can be dried in a continuous rotary dryer for use as a feed. Pilot-plant operation indicates that the process offers a profitable solution to the pear-canning industry's serious waste-disposal problem.

A PROCESS FOR THE RECOVERY OF SUGARS and other soluble solids from pear peelings, cores, and trimmings for use as a sirup base for canned pears has been described in a preliminary report (9). The steps in this process were adapted from conventional methods for treating fruit wastes, preparing fruit

juices, and refining sugar solutions. The steps were specifically selected and designed to provide as simple and economical a process as possible, consistent with the quality essential to a sirup base for canned pears. The present report describes a pilot plant application of this process and presents pilot plant

and laboratory data on several of the unit operations involved.

The recovery of sugars from fruit cannery wastes in a form suitable for use as a sirup base has been proposed by a number of workers. Such processes appear logical, as sugars are the main constituent of economic value in fruit