QUICK-CURING OF SUPERPHOSPHATE

Effect of Dryer Inlet Air Temperature, Phosphate Rock Particle Size, and Surfactants

G. L. BRIDGER[1] and JOHN L. KEARNS[2]

Department of Chemical and Mining Engineering, Iowa State College, Ames, Iowa

Laboratory studies were made on the effect of dryer inlet air temperature, phosphate rock particle size, and surfactants on the composition and physical properties of superphosphate made by a quick-curing process which consists of mixing phosphate rock with 55% sulfuric acid and drying in a Roto-Louvre dryer. Inlet air temperature had no effect on phosphorus pentoxide conversion, but a marked effect on the particle size of the product. Moisture content of the product was the most important factor affecting conversion. Decreasing rock particle size improved conversion, but excessive grinding was required to produce a significant improvement. Surfactants added during the mixing of the rock and acid did not improve conversion.

A QUICK-CURING PROCESS for normal superphosphate developed at Iowa State College (5) consists of acidulating phosphate rock, ground to approximately 60% −200 mesh, with 40 to 60% sulfuric acid in the proportion of 1.8 pounds of sulfuric acid (100% basis) per pound of phosphorus pentoxide in the rock, denning only long enough to allow the formation of a firm mass which can be easily disintegrated, and drying in a Roto-Louvre dryer. The previous laboratory study (5) gave a maximum of 93% phosphorus pentoxide conversion, whereas the conventional storage-curing process, which uses about 70% sulfuric acid, gives 97 to 99% conversion.

This study was undertaken to determine the effect of certain variables— dryer inlet air temperature, rock particle size, and use of surface active agents —on the composition and physical condition of the product, with the objective of improving conversion and effecting granulation of the quick-cured product.

Procedure

The phosphate rock used in this study was Florida land pebble rock. Its chemical composition and screen analysis are given in Tables I and II.

Calcium oxide, iron oxide, and aluminum oxide were determined by methods of the Association of Florida Phosphate Mining Chemists (7). Fluorine was determined by the method of Brabson, Smith, and Darrow (3). All other determinations were by methods of the Association of Official Agricultural Chemists (2).

The rock referred to as "standard" was typical of that used in industrial practice. Screen analysis was made on Tyler standard screens, the −150-mesh fraction being wet screened. Sample

P-1 was entirely wet screened, and was prepared by a single pass of the standard sample through a Braun pulverizer, Type AU. Samples P-2, P-3, and P-4 were ground in a Schutz-O'Neill internally air-classifying centrifugal impact pulverizer.

Acidulations were carried out in a stainless steel kitchen mixing vessel with a household Mixmaster for stirring. In acidulating, 600 grams of the phosphate rock were added to 655 grams of 55% c.p. sulfuric acid, and agitated for 2 minutes with the mixer blades turning at 225 r.p.m. An acid concentration of 55% was used instead of 50% as preferred in the previous work (5), because the lower concentration did not produce as satisfactory a cake in the den in pilot plant tests. Both rock and acid were at room temperature prior to mixing. The superphosphate was denned for about 20 hours in a $4^3/_4$-inch cubic wooden box, painted with Tygon.

In preparation for the drying operation, the material was removed from the denning box about 15 minutes before it was put into the dryer, and was rubbed through a $1/_4$-inch screen by hand in as gentle a manner as possible to avoid pastiness. From this screened material, before the dryer charge was weighed, the feed sample was taken for analysis. The dryer was brought to temperature 1 to 2 hours before charging. Two pounds of superphosphate constituted a dryer charge and the time at which the material was actually placed in the dryer determined zero time for the recorded observations. Immediately after the dryer was charged, thermometers for measuring the outlet air temperature and the product temperature were inserted. Inlet air temperature was measured by a thermometer in the air duct immediately ahead of the dryer. Diagrammatic views of the laboratory Roto-Louvre dryer used and the position of the thermometers are shown in the previous

study (5). An appreciable change in the volume of the bed occurred during the course of a run due to removal of samples. At the beginning, the bed was approximately 8 inches across the top and 2 inches deep. The depth at the end of the run was approximately $1^1/_4$ to $1^1/_2$ inches and the top width lessened proportionately.

Samples taken for analysis were kept in tightly sealed 1-ounce sample bottles. For analytical purposes, several grams were ground and 1-gram samples weighed for use. Processing of the samples was begun within 1 hour (in most cases within 15 minutes) of their withdrawal from the dryer. Moisture (100° C. oven method), citrate-soluble phosphorus pentoxide, and total phosphorus pentoxide were determined by methods of the Association of Official Agricultural Chemists (2), except that the phosphorus pentoxide in the solutions was determined by the colorimetric procedure of Bridger, Boylan, and Markey (4). Analytical determinations made immediately after drying were designated "0-day." The samples still tightly sealed were kept in a chamber held at a constant temperature of 85° F. and a constant relative humidity of 32% for 3 days, when they were analyzed again; these analyses were designated "3-day." In some cases additional

Table I. Composition of Florida Phosphate Rock (Dry Basis)

	%
CaO	47.4
MgO	0.40
F	4.6
P_2O_5	33.3
Fe_2O_3	1.06
Al_2O_3	1.33
SiO_2	8.96
Moisture[a]	0.53

[a] Wet basis.

unsealed samples were retained in the chamber for 3- and 30-day analyses.

The product samples showed a wide range of particle size variation, and this was determined by using Tyler standard screens with very gentle hand shaking.

Effect of Inlet Air Temperature

The range of inlet air temperature studied was that which would cover practical values for industrial operation and still not greatly exceed product temperatures indicated in the previous study (5) to be detrimental to the super-phosphate—namely, about 275° F. A constant inlet air temperature throughout each run, rather than a constant product temperature as in the previous study (5), was used to simulate the conditions that occur in a continuous dryer. Samples removed at varying time intervals from the laboratory batch dryer corresponded to samples from varying distances through a continuous dryer.

Figure 1 is a representative plot of the data for a single run in which the inlet air temperature was 800° F. It is from such plots that the compositions of products at 1 and 3% moisture in the subsequent tables are drawn. The basis for interpolation or extrapolation of the data was the moisture on zero day, and whatever drying time gave the required moisture on zero day was also the drying time at which the 3-day data were read from the plots.

In Figure 1 it is noteworthy that a distinct maximum occurs in the conversion curve for the third day. Throughout this series, the maximum conversion on the third day occurred within the moisture range of about 9 to 10%. Since the conversion of quick-cured superphosphate is approximately constant at any moisture content on zero day, the rate of increase of conversion during storage is much greater at this moisture content than at any other, with the exception of the undried feed material. The maximum conversion on the third day was as high as 99% (sealed storage), while an average sealed feed in the same period reached only 94 or 95%. On the average, unsealed feed reached 97% con-
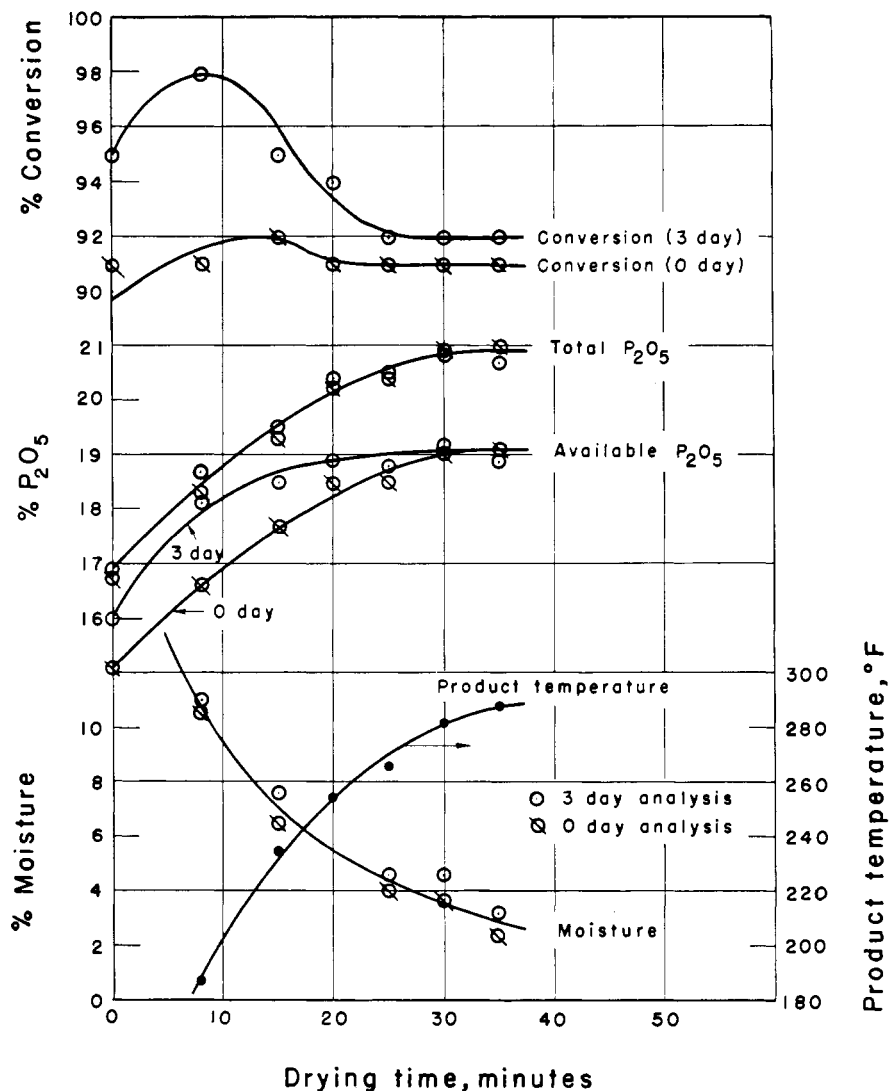
version, while its moisture content decreased to approximately 13% in the same period. The average figures for all runs of this series indicate that maximum conversion of the quick-cured superphosphate exceeds the unsealed feed conversion by 1 to 2%, and sealed feed conversion by 4 to 5% after 3 days' storage.

The total phosphorus pentoxide curves for this run at 0 and 3 days are almost

identical, as they should be, since the samples were kept in sealed bottles. However, in many of the runs this was not the case; possibly either gain or loss of moisture was caused by leakage. For this reason, the total and available phosphorus pentoxide contents of the 3-day samples were not comparable with those of the 0-day samples and are not reported. It is felt that the phosphorus pentoxide conversions are comparable, however, and these are reported.

Table III presents the results of the quick-curing experiments at various inlet air temperatures. Except for the fresh mix analyses and some of the conversions, the data are for zero day only. Interpolated values are shown for 1 and 3% moisture samples, which might be practical levels of drying in commercial production.

Figure 2 shows the effect of inlet air temperature on product composition. The conversion obtained at either the 3 or 1% moisture level on zero day was substantially the same regardless of inlet air temperature. On the third day, the conversion at the 1% moisture level

Figure 1.   Drying of superphosphate with inlet air temperature of 800° F.

Table II.   Screen Analyses of Phosphate Rock Samples

Screen Mesh	% of Sample				
	Standard	P-1	P-2	P-3	P-4
+ 65	5.6	1.4	. . .	. . .	. . .
− 65 +100	8.7	1.8	. . .	. . .	. . .
−100 +150	10.8	3.5	. . .	. . .	. . .
−150 +200	13.8	6.2	. . .	. . .	. . .
−200	61.1	. . .	. . .	. . .	. . .
−200 +325	. . .	13.4	. . .	. . .	. . .
−325	. . .	. . .	4.0	. . .	. . .
−325 +400	. . .	0.8	. . .	. . .	. . .
−325	. . .	. . .	96.0	. . .	. . .
−400	. . .	72.9	. . .	. . .	. . .
Maximum micron size	. . .	. . .	. . .	30–40	20–30

Table III. Effect of Inlet Air Temperature on Composition of Quick-Cured Superphosphate

(0-day analyses)

Sample No.	Drying Time, Min.	Prod. Temp., ° F.	Exit Air Temp., ° F	Moist., %	% P₂O₅ Total	% P₂O₅ Avail.	Conversion 0-day	Conversion 3-day
Inlet Air Temperature, 500° F.								
Mix	(−20 hours)a	...	...	20.2	17.2	10.5	61	..
Feed	0	...	...	20.4	18.9	...	..	94
1	20	174	176	11.9	18.2	16.2	89	97
2	40	226	218	8.1	18.8	16.8	90	95
3	60	244	230	5.3	19.5	17.5	90	93
4	75	253	240	3.4	20.0	18.2	91	93
	80b	256b	...	3.0b	20.1b	18.3b	91b	92b
5	90	258	244	2.2	20.4	18.6	91	92
6	120	263	248	1.2	21.0	19.3	92	92
	123c	264c	...	1.0c	21.1c	19.4c	92c	92c
Inlet Air Temperature, 600° F.								
Mix	(−21 hours)a	...	...	20.4	16.8	10.4	62	..
Feed	0	...	...	21.3	16.4	14.7	90	94
1	20	228	217	8.1	18.9	17.0	90	95
2	40	270	250	3.7	20.0	18.1	91	93
	45b	267b	...	3.0b	19.9b	18.1b	91b	93b
3	60	278	256	1.5	20.3	18.5	91	92
	63b	282b	...	1.0b	20.4b	18.6b	91b	92b
4	75	278	263	0.0	20.8	18.8	91	91
5	90	298	262	− 2.0	21.0	19.1	91	93
6	120	308	284	− 2.8	22.0	20.1	91	91
Inlet Air Temperature, 700° F.								
Mix	(−21 hours)a	...	...	21.8	18.8	12.6	67	..
Feed	0	...	...	19.1	17.6	15.7	90	95
1	10	190	197	10.0	18.9	17.1	91	99
2	15	210	224	8.2	19.2	17.4	91	98
3	20	248	243	6.8	19.6	18.0	92	96
4	25	264	254	4.3	20.5	18.5	91	94
	27b	274b	...	3.0b	20.7b	18.7b	91b	93b
5	31	284	268	1.5	21.0	19.0	90	92
	33b	286b	...	1.0b	21.1b	19.1b	91b	92b
6	40	295	278	− 0.4	21.5	19.4	90	91
Inlet Air Temperature, 800° F.								
Mix	(−21 hours)a	...	...	22.5	16.6	10.2	62	..
Feed	0	...	...	22.6	16.7	15.2	91	95
1	8	187	181	10.5	18.3	16.6	91	98
2	15	235	196	6.5	19.3	17.7	92	95
3	20	255	200	5.2	20.4	18.5	91	94
4	25	266	211	4.0	20.5	18.5	91	92
5	30	282	224	3.6	20.9	19.0	91	92
	31b	282b	...	3.0b	20.8b	19.0b	91b	92b
6	35	288	238	2.2	21.1	19.1	91	92
	47c	...	...	1.0c	21.0c	19.2c	91c	92c
Inlet Air Temperature, 900° F.								
Mix	(−20 hours)a	...	...	22.4	16.6	10.9	66	..
Feed	0	...	...	21.5	16.5	14.7	90	93
1	8	193	195	8.6	18.5	16.7	91	97
2	15	253	224	4.0	22.0	20.4	92	93
	17b	264b	...	3.0b	20.2b	18.2b	92b	92b
3	20	278	231	1.8	18.4	16.7	90	92
	21b	286b	...	1.0b	20.8b	18.7b	91b	92b
4	25	298	246	− 0.3	21.3	19.3	91	92
5	30	303	268	− 1.5	21.3	19.0	90	91
6	35	311	277	− 1.1	21.6	19.5	90	92
Inlet Air Temperature, 1000° F.								
Mix	(−23 hours)a	...	...	22.3	16.8	10.4	62	..
Feed	0	...	...	20.4	16.8	15.3	91	95
1	3	191	199	16.7	17.2	15.2	89	91
2	6	194	201	10.6	18.8	17.1	91	98
3	9	214	208	6.2	19.6	17.9	91	95
4	12	237	218	3.6	19.9	17.9	90	94
	13b	254b	...	3.0b	20.0b	18.3b	91b	93b
5	15	265	219	2.4	20.1	18.4	92	97
	17b	268b	...	1.0b	20.5b	18.6b	91b	92b
6	18	268	219	0.7	20.7	18.8	91	91

a Analysis begun within 15 minutes of acidulation.
b Interpolated values.
c Extrapolated values.

had increased by about 1% and at the 3% moisture level by about 1.5%.

Total and available phosphorus pentoxide contents showed a slight maximum in the range 700° to 800° F.—probably because product temperatures were at a maximum in this range, and the loss of water of constitution was somewhat greater. The dotted curves indicate the estimated available phosphorus pentoxide content for 1 and 3% moisture after 3-day curing. Maximum available phosphorus pentoxide content would be achieved with an inlet air temperature of 750° to 800° F.

The fact that maximum product temperature for a given moisture content was reached at an intermediate inlet air temperature can be explained by a consideration of time-moisture-temperature relationships. Figure 3 shows the product temperature for 1 and 3% moisture levels, and drying times to reach these levels. Table IV shows data at the various inlet air temperatures for drying a given time (10 minutes), to a given product temperature (250° F.), and to a given moisture content (3%). It is apparent that at the highest inlet air temperatures used, the moisture content was reduced to the given level before there was time for the higher product temperatures to be reached.

The effect of moisture content of the 0-day and 3-day samples on conversion is shown in Figure 4, which includes data for all the runs with inlet air temperatures from 500° to 1000° F. and product temperatures from 174° to 311° F. Despite the wide temperature range represented, excellent correlation of conversion with moisture content resulted, indicating that moisture content is the controlling variable. Conversion is constant at about 91% over the entire range of moisture content for the 0-day samples. On sealed storage for 3 days, however, a maximum conversion of 98 to 99% develops at a moisture content of 9 to 10%. At lower moisture contents the increase in conversion on 3-day storage decreases, and is only about 1% at very low moisture contents.

From a practical standpoint, the optimum moisture content would depend on the use to which the superphosphate is put. For direct application, it would probably be best to dry the superphosphate to a moisture content in the range 1 to 6%, since storage and handling properties of such a product would be satisfactory, and available phosphorus pentoxide content would be greater, despite lower conversion. For ammoniation and mixed fertilizer manufacture, drying to 9 or 10% and storing for a few days before use would be preferable, as the higher moisture content would promote ammonia absorption and maximum conversion would be obtained. Such a product would also be suitable as a phosphatic raw material

for mixed fertilizer processes employing a slurry-mixing step, such as the Martenet process (7), since a dry superphosphate is not needed and a high moisture content would be helpful in developing the slurry.

Table V shows the composition of superphosphates made with 55 and 72% sulfuric acid at various times after mixing under conditions of both open and sealed storage; none of them was dried in the Roto-Louvre dryer. For maximum conversion and low moisture content, 30 days of storage curing is necessary. Moderately high conversion is attained on 3-day sealed storage, but moisture content is high under these conditions. The benefit of using 55% acid in achieving higher conversion is apparent.

Figure 5 shows samples of the products made at various inlet air temperatures, and Table VI shows their screen analyses. A wide variation in particle size with temperature was observed. There was also a marked difference in the physical appearance of the particles. Those formed at the lower inlet air temperatures had a hard, thick, impervious-appearing surface coating of the order of $^1/_{16}$ to $^1/_8$ inch in thickness in particles ranging from $^1/_2$ to $^3/_4$ inch in diameter. At higher inlet air temperature the impervious-appearing layer was not observed and pores similar in appearance to those in the interior of the particle were noted on the surface. Table VI contains a screen analysis of the product of a run with 400° F. inlet air temperature. No other data for this run are reported because the particle size was much too large either for practical purposes or for obtaining representative samples for analytical determination.

During the drying process, material dried with a low inlet air temperature maintained a very wet appearance on the surface of the particles for about 30 minutes with the lowest temperature, while the particles grew larger. With the highest inlet air temperature, some agglomeration took place in less than a minute, but the wet appearance of the particles was not observed. The relation between inlet air temperature and drying time to reach a given moisture content is interesting in relation to the physical appearance of the particles. The time required decreases markedly when the inlet air temperature reaches about 750° F., the point of maximum product temperature, and the point at which pores began to appear on the surface of the particles.

Effect of Rock Particle Size

It was hoped that the use of phosphate rock somewhat finer in particle size than ordinarily used would improve conversion in the quick-curing process, and consequently tests were made with rock ground to the various particle sizes shown in Table II. The same
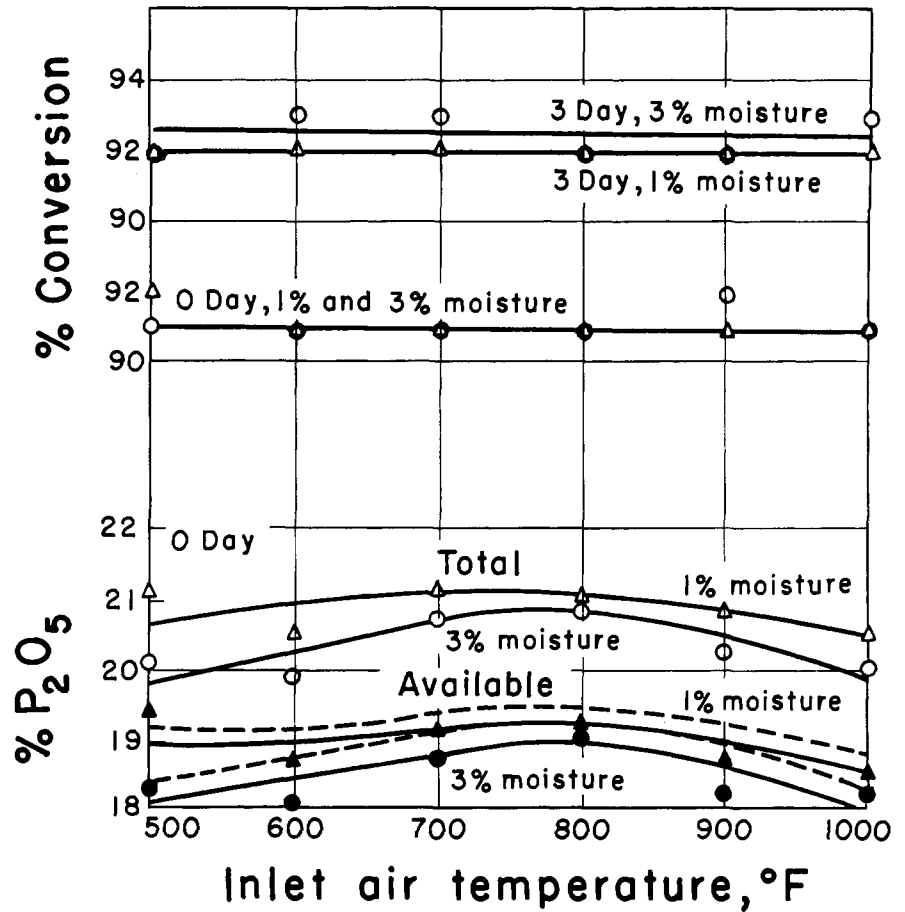

Figure 2. Effect of inlet air temperature on composition of quick-cured superphosphate
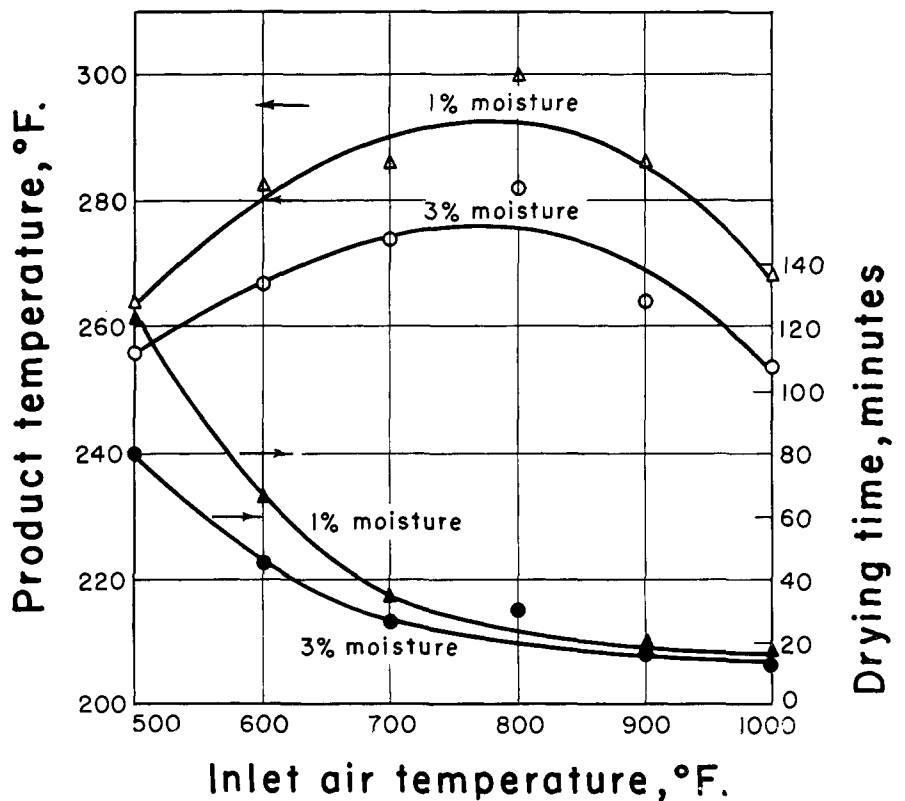

Figure 3. Effect of inlet air temperature on product temperature and drying time

Table IV. Moisture-Time-Temperature Relationships at Various Inlet Air Temperatures

Inlet Air Temp., ° F.	10-Min. Drying		Prod. Temp., 250° F.		3% Moisture[a]	
	Prod. temp., ° F.	Moist., %	Drying time, min.	Moist., %	Prod. temp., ° F.	Drying time, min.
500	. . .	. . .	71	4.0	256	80
600	. . .	. . .	31	5.4	267	46
700	190	10.0	21	6.4	274	27
800	205	8.6	19	5.3	271	20
900	212	7.2	15	4.3	269	16
1000	218	5.4	13	3.2	254	14

[a] Taken from Figure 3.

Table V. Composition of Fresh and Storage-Cured Superphosphates

Sample	Storage	Age, Days	Moist., %	% P₂O₅ Total	Avail.	Conv.
			Made with 55% H₂SO₄			
Mix	None	−20 hr.[a]	21.6	17.0	10.7	63
Feed	Den	0	21.1	16.8	15.1	90
Feed	Sealed	3	21.5	16.7	15.7	94
Feed[b]	Open	30	3.6	19.9	19.7	99
			Made with 72% H₂SO₄			
Mix	None	−20 hr.[a]	13.0	18.2	11.9	65
Feed[b]	Den	0	11.8	19.7	16.2	82
Feed	Sealed	3	11.2	19.5	17.7	91
Feed[b]	Open	30	2.5	20.6	19.8	96

Note: In the header, the "% P₂O₅" spans the Total, Avail., Conv. columns.

[a] 15 minutes after mixing.
[b] From Bridger and Kapusta (5).

Table VI. Screen Analyses of Products Made at Various Inlet Air Temperatures

Size	Inlet Air Temperature, ° F.						
	400	500	600	700	800	900	1000
+0.742 inch	40.9	. . .	. . .	. . .	. . .	. . .	. . .
+0.525 inch	73.7	. . .	. . .	. . .	. . .	. . .	. . .
Mesh							
+3	90.4	64.7	33.3	3.6	. . .	. . .	. . .
+4	98.1	88.2	62.1	41.6	. . .	. . .	. . .
+6	99.6	96.9	81.1	81.0	5.0	4.0	2.0
−6	0.4	. . .	. . .	. . .	. . .	. . .	. . .
+8	. . .	98.5	88.6	96.5	. . .	. . .	. . .
−8	. . .	2.4	. . .	. . .	. . .	. . .	. . .
+10	. . .	. . .	92.6	99.6	22.2	33.6	23.8
−10	. . .	. . .	7.6	0.4	. . .	. . .	. . .
+12	. . .	. . .	. . .	. . .	35.2	52.0	39.8
+14	. . .	. . .	. . .	. . .	52.8	70.2	58.0
+16	. . .	. . .	. . .	. . .	75.0	87.6	72.6
−16	. . .	. . .	. . .	. . .	24.6	12.4	27.4

Table VII. Effect of Rock Particle Size on Conversion

Rock No.	Rock Size	Drying Time, Min.[a]	Prod. Temp., ° F.[a]	% P₂O₅[a] Total	Avail.	Conversion
		0-Day Analyses				
Standard	61% −200 mesh	91	238	20.2	18.4	91
P-1	80% −325 mesh	67	204	19.8	18.2	92
P-2	96% −325 mesh	52	188	20.4	18.9	93
P-3	30–40μ	69	187	19.7	18.9	97
P-4	20–30μ	48	171	19.0	18.5	96
		3-Day Analyses				
Standard	61% −200 mesh	. .	. . .	19.8	18.4	93
P-1	80% −325 mesh	. .	. . .	20.1	18.6	94
P-2	96% −325 mesh	. .	. . .	20.6	19.6	96
P-3	30–40μ	. .	. . .	19.8	19.4	97
P-4	20–30μ	. .	. . .	19.4	18.9	99

[a] For 6.0% moisture content of 0-day sample.

procedure as in the inlet air temperature study was used, with the exception of mixing the rock and acid and denning time. It was necessary to pour the two reactants simultaneously and uniformly into the mixing vessel, because the reaction time was much shorter for the fine rocks and otherwise good mixing could not be obtained before solidification of the mass took place. The mixture was denned for 12.5 hours. An inlet air temperature of 600° F. was used.

The results of the tests are shown in Table VII. Compositions of the products at 6.0% moisture are shown after both 0- and 3-day sealed storage. As rock particle size decreased, the drying time to reach a given moisture content also decreased, and the product temperature was lower; this was doubtless due to the more friable nature of the superphosphate made with the finer rock. Only the two finest rock samples (P-3 and P-4), in the 20- to 40-micron range, showed marked improvement in conversion immediately after drying. After 3 days, the P-2 sample, which was essentially all −325 mesh, also showed a moderate improvement. However, to obtain a significant improvement in conversion over that obtained with a rock of the usual commercial particle size, the additional grinding required would be excessively expensive.

Use of Surfactants

An exploratory series of tests was made to determine whether addition of a small amount of surface active agent to the superphosphate during mixing would improve conversion in the quick-curing process. Thirteen different commercial surface active agents were tried, including anionic, cationic, and nonionic types. The agents were added to the 55% sulfuric acid before mixing in the proportion of 1 pound of active ingredient per ton of superphosphate. An inlet air temperature of 600° F. was used for drying.

No significant increase in phosphorus pentoxide conversion was found for any of the surface active agents, and it is concluded that they have no advantage in the quick-curing process. Details of these tests are on file in the Iowa State College Library (6).

Conclusions

Dryer inlet air temperature does not affect conversion of quick-cured superphosphate over the range 500° to 1000° F., but it markedly affects particle size of product. With inlet air at 700° F. a product essentially all −3 +10 mesh was made, and with 900° F. air, a −6 +16 mesh product was made. The available phosphorus pentoxide content of superphosphate is slightly

higher at inlet air temperatures in the range 750° to 800° F. than at other temperatures.

The moisture content to which the superphosphate was dried did not affect conversion of the product immediately after drying, but was the controlling factor in conversion after 3-day storage. A maximum conversion of 99% was obtained at a moisture content of 9.5% after 3-day storage. The maximum available phosphorus pentoxide content of the product, however, was obtained by drying to 1% moisture with a conversion of 92%. For ammoniation and manufacture of mixed fertilizer the product with maximum conversion and higher moisture content should be preferable, whereas for direct application the product with higher available phosphorus pentoxide content and lower moisture content should be preferable.

Phosphate rock ground to a very much finer particle size than normally used for superphosphate manufacture resulted in improved conversion, but the degree of grinding required would not be economically feasible.

Of 13 surfactants of various types, none resulted in improved conversion.
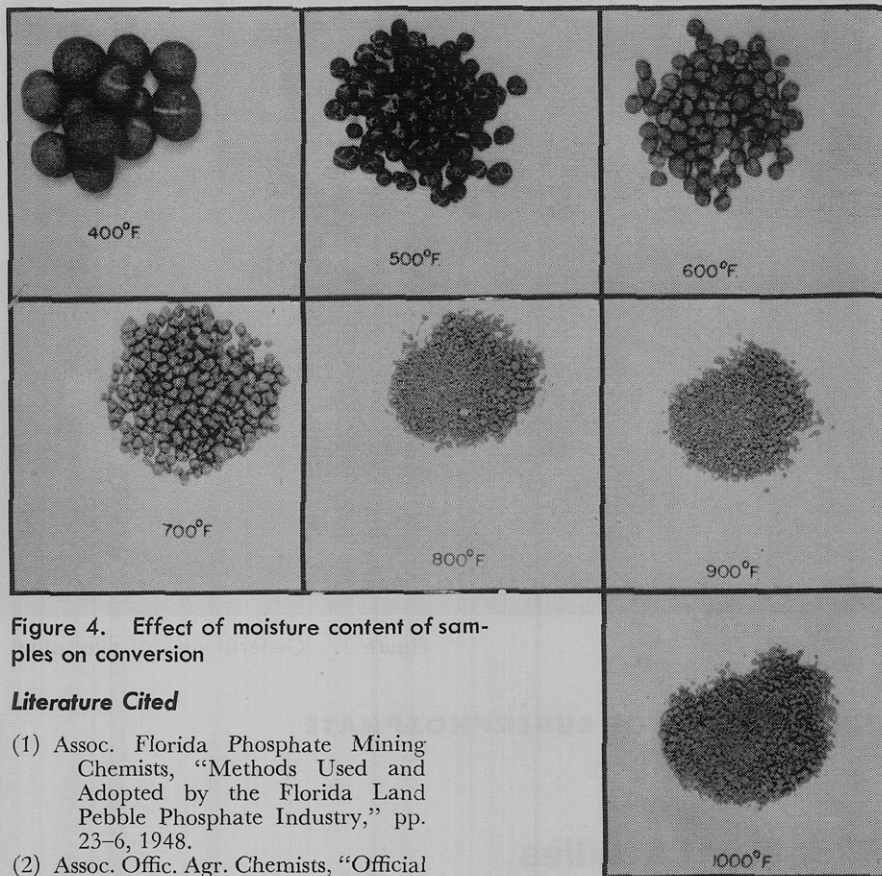
Acknowledgment

Figure 4. Effect of moisture content of samples on conversion

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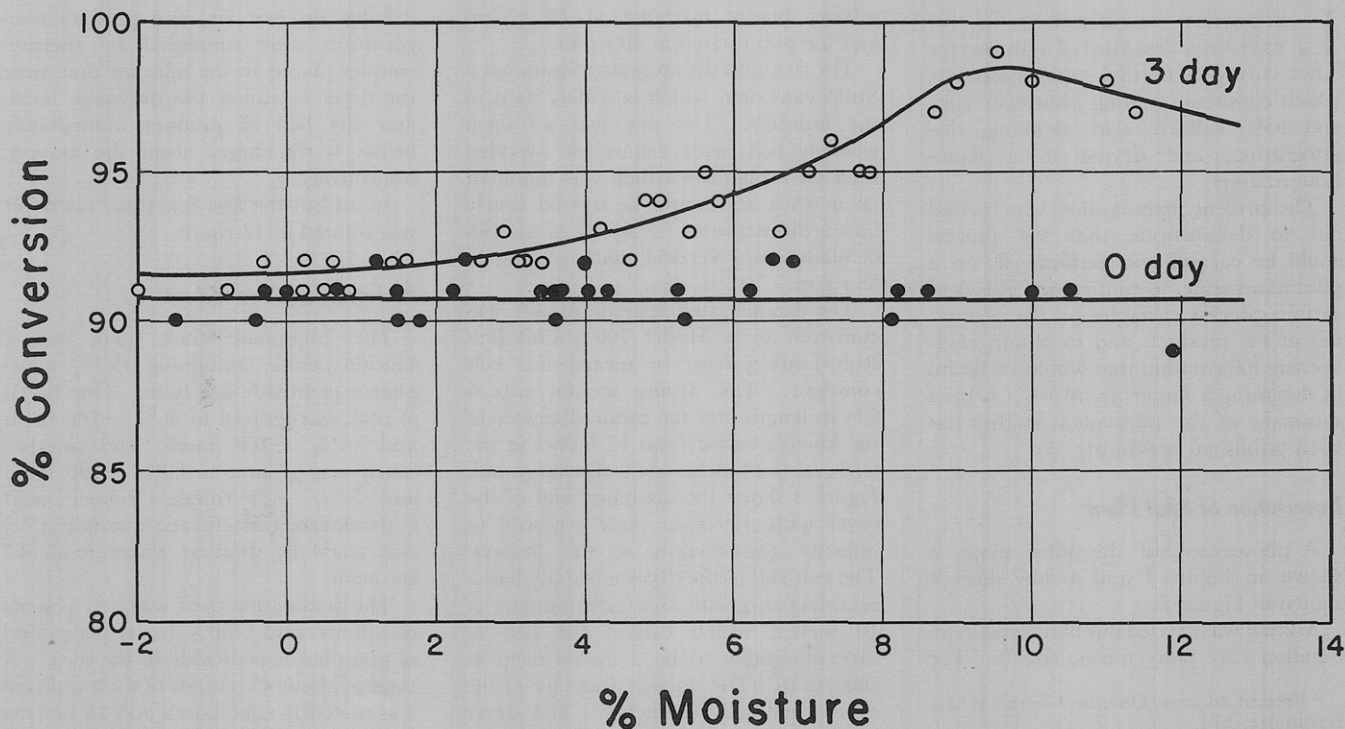(7) Martenet, S. J., *Agr. Chemicals* **9,** 46–8, 138–41 (1954).

Figure 5. Samples of quick-cured superphosphates made at various inlet air temperatures