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LOSS OF WATER SOLUBLE PHOSPHATES DURING GRANULATION
OF SUPERPHOSPHATES

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In Great Britain, the sales value of phosphate, either in the form of superphosphate, triple superphosphate or in compound fertilizers, is based on a test for the amount of material soluble in water. In effect, this means the sum of the mono-calcium phosphate and free phosphoric acid contents together with any meta- or pyrophosphates which may be formed during, for example, a granulation process in which the phosphate material is subjected to an elevated temperature. The determination of the latter two salts is ensured in the official method of analysis by hydrolysis to the ortho- form prior to precipitation.

In addition, during the granulation of compound fertilizers in which the source of phosphate is admixed with ammonium and potassium salts, it can be visualized that reaction with free phosphoric acid or double decomposition with phosphate salts, may result in ammonium and potassium phosphate formation. The free phosphoric acid may intentionally be made to react initially with aqueous or gaseous ammonia to produce ammonium phosphate. The effect of such reactions on the ratio of water soluble phosphoric acid to total phosphoric acid originally present has not previously been evaluated commercially.

A statement of analysis must be given with any fertilizer sold in Great Britain. In the case of the fertilizer types mentioned above, the statutory requirements necessitate a statement in respect of both water soluble and water insoluble phosphoric acid contents. Both of these are expressed in terms of "P₂O₅" and a small variation is allowed in both cases.

The results of the reactions between phosphate rock and sulphuric acid are sufficiently well known, at any given strength of acid and for any given reaction conditions, to allow an accurate estimate of the expected conversion* to be made.

* In this paper, the term "conversion" is used to signify the percentage relationship of the water soluble phosphoric acid content of the material to its total phosphoric acid content.

The majority of the water soluble phosphoric acid made by Messrs. Fisons Limited is sold in the form of granulated material, either as a straight superphosphate or as compound fertilizers. A study of the routine analytical results of the products sold suggested that a reduction in the conversion of the source of phosphate was occurring during granulation. This not only implies a financial loss, (since costly acid has been used to produce water soluble material), but it necessitates an allowance in the formulation of a compound fertilizer in order that no large part of the variations permitted under the official Regulations are absorbed. At about the same time, it was decided to investigate the effect on conversion if the moisture content (normally 2 $\frac{1}{2}$ -3%) of granular compound fertilizers was lowered to a maximum of 1%.

Accordingly, a detailed sampling and analytical programme was laid down to evaluate any such losses of water soluble phosphoric acid which might occur during granulation.

This paper does not set out to present a chemical explanation of the reactions which may occur. It presents, in summarised version, the results of a series of tests carried out on seven plants under a variety of conditions.

GRANULATING PLANT CIRCUITS

The experiment was complicated in the first place by the fact that in the plants chosen for the test, four different circuits were in operation. Two of the four circuits were basically the same. These differences in circuits could be expected to exert a large influence on loss of conversion if it was in any way due to the heat treatments given to the material. It is therefore necessary to bear in mind the circuit employed during consideration of the results obtained.

Circuit A. After the material has left the drier, it is screened to remove oversize. The material passing the screen is cooled and rescreened to provide product and fines fractions. The oversize is cracked and rescreened; material passing the screen is added to the product fraction while oversize is again recrushed. The fines fraction is returned, either to the batch mixer handling incoming raw material (Circuit A₁), or to the granulator where the mixed raw materials are sprayed with water and rolled to form granules (Circuit A₂).

Circuit B. Oversize is similarly removed from material leaving the drier; after crushing it is fed back to the drier and redried. The fines are removed from the stream leaving the cooler and are passed to the granulator.

Circuit C. In this circuit, the oversize removed from the stream leaving the drier is completely crushed until it is below product size and it is then recycled to the batch mixer, together with the fines, and incorporated into incoming raw material.

In all circuits, fertilizer dust removed from the cyclones handling the drier and cooler gas streams is returned with the fines.

The size of the driers concerned relative to the tonnage throughput varied, but the retention time in all driers was 25 minutes \pm 4 minutes.

The volume of the gas through the driers was, for various reasons, not recorded, and it is probable that no constant ratio of throughput to gas volume exists for the seven plants over which these results are based.

TYPES OF GRANULATION

In attempting to produce a compound fertilizer of lower moisture content, there were several alternatives open to us. They all required modification of our normal granulation procedure. Such modifications were tested in comparison with normal methods of granulation on each plant and are as follows:-

1. The inlet gas temperature was raised to as high a temperature as possible without causing undue damage to plant and the throughput reduced so that the end product had a moisture content of 1% or less.
2. A process alteration allowed us to reduce appreciably the total amount of water necessary to effect the same degree of granulation. In this way, we were able to maintain output ratios comparable with normal operation and still achieve a moisture content of less than 1%, although the temperature conditions of the inlet gas and of the granules leaving the drier were similar to No. 1.
3. Normally produced material was not cooled, but was subjected to a second drying period with an inlet gas temperature controlled so that the temperature of the material leaving the second drier was no higher than it had been on leaving the first drier. In both driers, hot gases passed concurrently with the material.

METHOD OF EVALUATION OF THE LOSSES

The same compound fertilizer was produced on all plants. A combination of the circuits and methods of granulation discussed above was employed.

The compound selected analysed:-

Nitrogen	9.0%
Water soluble P_2O_5	8.25%
Insoluble P_2O_5	0.75%
Potash	15.0%

and derived its phosphate partly from superphosphate and partly from triple superphosphate. Sulphate of ammonia provided the nitrogen, while potash came from raw muriate. There was no insoluble P_2O_5 from any third ingredient, so we could dismiss errors due to disproportionate mixing.

Samples of raw material were taken at regular intervals during the test and analysed for water soluble P_2O_5 and total P_2O_5 . In this way, the conversions for each material could be calculated. A knowledge of the relative amounts of single superphosphate and triple superphosphate in the mixture enabled an "expected" conversion for the compound to be calculated. The conversions of the superphosphate and triple superphosphate used as raw materials are so nearly equal that any effects due to disproportionate mixing will be small. For each test the mean of the "expected" conversion was calculated and, in addition, statistically, its variance and standard error. Similar calculations were made for the "achieved" conversion based on water soluble and total P_2O_5 determinations carried out on samples of the granular compound fertilizer produced.

If the variances of the "expected" and "achieved" mean conversions were not shown to be different by a statistical test of significance ("F"-test; $P < 5\%$), then the means were compared by Student's "t"-test.

This procedure was followed in all cases and the results of such tests have been summarised, together with relevant plant data, in Table I. Where a significant loss of conversion has been detected, this has been quoted as a percentage of that originally present in the raw material.

Due to the deleterious effect on paper bags in storage, it is not usual, in Great Britain, to dry straight superphosphates to a low moisture content. However, some sampling of raw materials and product was carried out during the granulation of single superphosphate made from both Morocco and Nauru rocks. Such analytical results have been treated in the same manner and are shown in Table II.

In both Tables I and II, columns have been added which represent, empirically, the drier load.

The linear factor is defined as the:
$$\frac{\text{throughput in tons/hour} \times 1000}{\text{drier diam. in ft.} \times \text{drier length in ft.}}$$

The volume factor is defined as the:
$$\frac{\text{throughput in tons/hour} \times 10,000}{(\text{drier diam. in ft.})^2 \times \text{drier length in ft.}}$$

The lower the value, the less highly is the drier loaded.

DISCUSSION OF RESULTS

1. During normal granulation in which the temperature of granules leaving the drier is $100^{\circ}\text{C} \pm 5^{\circ}\text{C}$, the drier loading does not appear to be directly related to the loss of conversion.
2. For a given circuit, on normal granulation, the higher the inlet gas temperature, the higher the loss of water soluble P_2O_5 .
3. For a given method of granulation and similar operating conditions, there is little difference in losses between circuits A₁, A₂ and B. However, circuit C shows greater losses. This can be expected from the theoretical consideration that a greater percentage of the fertilizer material is subjected to more prolonged heating during its processing when this circuit is in operation.
4. For comparable plant operating conditions, the loss increases as the moisture content of the granules decreases and as the temperature of the granules ex drier, increases.
5. For a given circuit, granulating methods 1 and 2 give similar losses while No. 3 is greater even if the temperature of the granules ex drier remains low. Hence the length of time during which a granule remains at an elevated temperature is important and emphasises the need for rapid cooling immediately after the required degree of dryness is reached. This is confirmed by the comparatively lower losses in the two plants operating on circuit B, which had the shortest retention time of all seven plants.

We can conclude, therefore, from the tests, that the loss of water soluble phosphate during normal granulation is proportional to:-

- (a) Drier retention time,
- (b) Drier inlet gas temperature,
- (c) Exit temperature of granules,
- (d) Moisture content of granules,

and that the choice of circuit and the method of granulation are important.

We can also estimate that normal granulation procedure, as operated by Messrs. Fisons Limited, results in the loss of 2% water soluble P_2O_5 in producing granular compounds containing $2\frac{1}{2}$ -3% moisture content.

If it is desired to lower the moisture content to less than 1%, then an additional 2% loss of water solubility will result.

COST OF THE LOSS OF WATER SOLUBILITY

Based on Messrs. Fisons Limited expected sales in the current fertilizer year, we estimate that in selling water soluble P_2O_5 obtained from single and triple superphosphate in granular form, the financial loss will amount to £20 for every £1,000 sales value.

If compounds are made and dried to less than 1% moisture, the additional financial loss amounts to £19.575 for every £1,000 sales value of the water soluble P_2O_5 contained in the compounds.

My thanks are due to the Directors of Fisons Limited for their permission to publish this paper, and to my colleagues in the factories, who have undertaken the not inconsiderable amount of sampling and analytical work necessary.

TABLE II
GRANULATION OF SINGLE SUPERPHOSPHATE

WORKS	CIRCUIT	TYPE OF GRANULATION	THROUGH PUT TONS/HOUR	MOISTURE CONTENT PRODUCT %	EXPECTED CONVERSION			ACHIEVED CONVERSION			"t" TEST BETWEEN EXPECTED AND ACHIEVED MEANS	ESTIMATE OF LOSS OF CONVERSION %	GRANULE TEMP. EX DRIER °C	GAS TEMP. INLET TO DRIER °C	DRIER LOADING		REMARKS
					NO. OF SAMPLES	MEAN	S. E.	NO. OF SAMPLES	MEAN	S. E.					LIN.	VOL	
1	A ₂	Normal	14.0	2.2	6	91.16	0.687	6	87.85	0.592	Significant	3.6	82	680	24.6	26.8	Nauru super approx. 24.0% total P ₂ O ₅
2	C	Normal	15.0	5.01	13	95.87	0.198	13	95.31	0.102	Significant	0.6	89	Not recorded	31.0	33.8	Morocco super approx. 19.0% total P ₂ O ₅
7	A ₁	Normal	8.5	7.06	12	97.07	0.262	12	97.17	0.244	Not significant	Nil	86	560	32.4	49.2	Morocco super approx. 19.0% total P ₂ O ₅

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TABLE I

GRANULATION OF COMPOUND FERTILIZERS

WORKS	CIRCUIT	TYPE OF GRANULATION	THROUGH PUT TONS/HOUR	MOISTURE CONTENT PRODUCT %	EXPECTED CONVERSION			ACHIEVED CONVERSION			"t" TEST BETWEEN EXPECTED AND ACHIEVED MEANS	ESTIMATE OF LOSS OF CONVERSION %	GRANULE TEMP. EX DRIER °C	GAS TEMP. INLET TO DRIER °C	DRIER LOADING		REMARKS			
					NO. OF SAMPLES	MEAN	S.E.	NO. OF SAMPLES	MEAN	S.E.					LIN.	VOL.				
1	A ₂	Normal	12.0	2.2	4	92.90	0.825	13	91.44	0.648	Not significant	1.8	100	600	21.1	23.0	High variation in analysis of raw supers and small number of samples.			
		1	10.5	0.5	7	92.09	0.543	24	84.26	0.904	Significant	8.5	140	700	18.5	20.2				
2	C	Normal	14.7	2.85	6	94.15	0.221	12	90.56	0.433	Significant	3.8	96	437	30.4	35.7				
		1	12.3	1.08	6	94.32	0.199	12	91.14	0.548	Significant	3.4	144	515	28.4	29.9				
		1	12.3	1.08	8	94.56	0.289	16	89.25	0.356	Significant	5.7	161	515	28.4	29.9				
3	B	1	7.5	0.5	30	93.32	0.157	30	92.29	0.297	Significant	1.1	140	Not (1) recorded	23.1	35.5	(1) Probably about 600°C			
4	B	Normal	8.25	2.5	No raw material anal.			4	92.75	0.321	Significant (2)	2.1 (2)	84	446	25.4	39.1	(2) Assuming that there was no change in raw materials, this represents a 2.1% additional loss of conversion during granulation by Modification 1.			
		1	6.3	0.7	No raw material anal.			5	90.25	0.401					125	523		19.7	39.1	
5	C	Normal	16.0	4.4	3	96.15	0.515	9	93.63	0.181	Significant	1.9 (3)	95	465	33.0	38.8	(3) Combining two sets of figures for normal granulation, reversion is equivalent to 1.9%			
		Normal	16.0	4.4	3	96.14	0.228	8	95.12	0.178					Significant	95		465	33.0	38.8
		1	14.8	1.5	10	96.18	0.216	26	92.34	0.270					Significant	4.0		138	554	30.6
6	A ₂	Normal	16.9	2.4	4	94.35	0.246	5	90.72	0.372	Significant	3.85	100	Not (4) recorded	34.8	41.0	(4) Probably about 550-600°C			
		1	14.2	0.3				7	87.73	0.676	Significant	7.0	144		29.4	34.5				
5	A ₁	1	16.2	0.85	9	94.80	0.275	36	91.99	0.163	Significant	3.0	132	582	33.4	39.2	The achieved means for modifications 1 and 2 are not significantly different from each other.			
		2	16.1	0.83	9	94.95	0.141	36	92.57	0.218	Significant	2.5	135	582	33.3	39.1				
5	A ₁	Normal	19.1	3.30	8	94.00	0.140	23	94.13	0.204	Not significant	Nil	97	595	39.5	46.5				
		2	18.1	1.13	8	93.97	0.198	24	90.97	0.287	Significant	3.2	130	533	37.3	43.9				
6	See Note (5)	3	16.0	2.3	11	95.11	0.148	22	92.44	0.256	Significant	2.7	106	Not (6) recorded	29.1	30.6	(5) Material ex 1st drier screened to remove fines. Product + oversize transferred to 2nd drier, after which oversize removed, crushed, in-grade material added to product. (6) Probably 550-600°C			
			14.0	0.43	11	95.11	0.148	8	90.19	0.591	Significant	5.2	109		375	29.6		34.8		