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TECHNICAL MEETINGS - STOCKHOLM

THE INCORPORATION OF AMMONIUM PHOSPHATES INTO GRANULAR NPK FERTILISERS

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1. INTRODUCTION

It is the purpose of this paper to describe some of the ways in which ammonium phosphates can be incorporated into granular NPK fertilisers, with particular reference to the use of conventional plants. In plants of this type the dry raw materials are batch weighed into the process and after a short period of mixing with recycled fines they are granulated by the addition of water in either a rotary tube or rotating pan. The material now in the form of damp granules (usually 10-15% moisture) is fed into a co-current rotary drier together with crushed oversize from a previous cycle. The discharge from the drier is then cooled and passed to the screens. All material in the correct size range is removed as product, and the fines, and oversize after crushing, are returned to the process.

In the post-war years the advantages of high plant food content fertilisers have become increasingly appreciated, and the ammonium phosphates with their 60+ plant food content are being increasingly used. In the past their use in fertilisers was restricted to a few large manufacturers, but today their increasing availability as an alternative raw material to superphosphate makes experiments on their incorporation of wide interest.

PROPERTIES OF THE AMMONIUM PHOSPHATES

There are two ammonium phosphates available to the fertiliser manufacturer, monoammonium phosphate (MAP) and the so-called diammonium phosphate (DAP) - a mixture of the mono- and di- salts, and of N:P atomic ratio usually 1.6 to 1.8. When produced for fertilisers, the analysis of the MAP is approximately 11½% N and 54½% P_2O_5, and the DAP about 18% N and 48% P_2O_5.

The properties of particular interest to the fertiliser manufacturer are as follows:-

(i) The high concentration in terms of plant foods which leads to savings in handling, packaging, transport, etc.

(ii) The ammonium phosphates, unlike superphosphates, are relatively pure crystalline chemical compounds. They do not have the thixotropic and binding properties of superphosphate which make for easy granulation; in consequence the amount of water required for granulation may be higher per ton of product though not necessarily per ton of plant food produced.

(iii) Monoammonium phosphate in particular has a steep solubility curve (approx. 23 g./100 ml. water at 0°C. and 173 g./100 ml. water at 100°C.), and care is necessary when drying fertilisers based on this material if build-up in the drier is to be avoided.

(iv) Diammonium phosphate decomposes to monoammonium phosphate when dried at temperatures above about 80°C., and a considerable amount of ammonia may be evolved.

(v) Fertilisers based on ammonium phosphates usually set in storage at a lower moisture content than do those based on superphosphate. This is as would be expected from the relative humidity v. moisture graph shown below - Fig. 1.

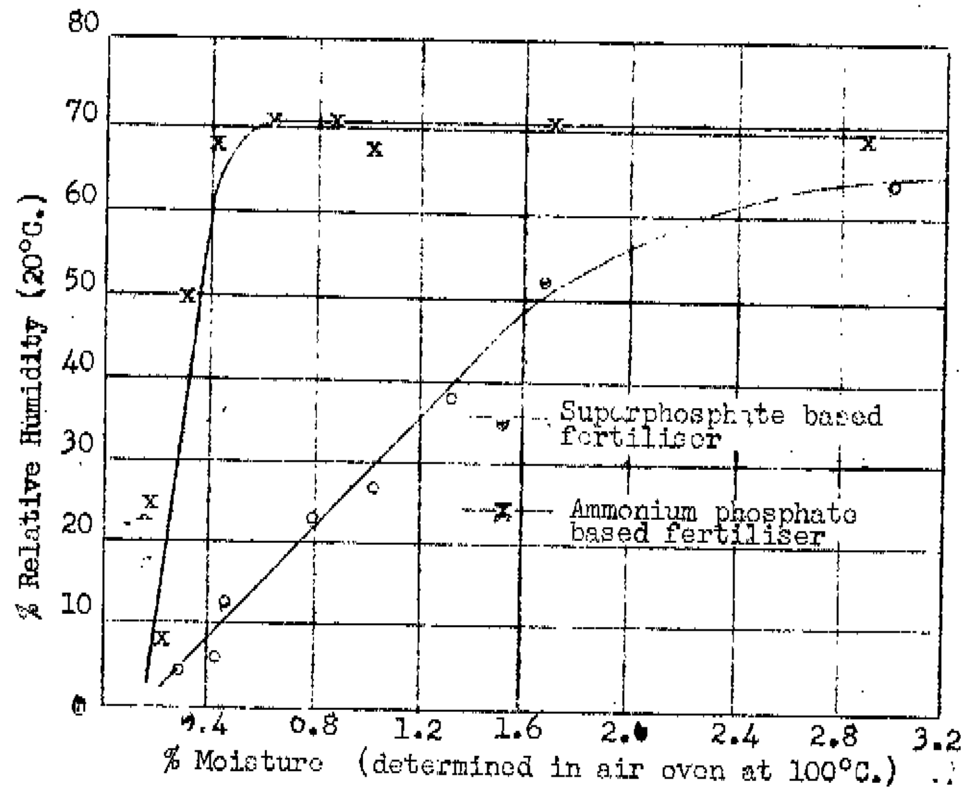

Fig. 1
Graphs of relative humidity against moisture content

The importance of relative humidity on caking behaviour has been discussed by B. Raistrick[1]. It has been the experience of our Company that most fertilisers will set on storage unless dried to that stage where their relative humidity is below about 30%, and while this level can be reached by drying superphosphate based fertilisers to about 0.8 to 1.6% moisture, it is normally necessary to dry those based on ammonium phosphate to about 0.2% moisture to obtain similar results.

(vi) Diammonium phosphate when mixed with superphosphate in sufficient amount will revert a proportion of the soluble P_2O_5 to the water insoluble form - a disadvantage in those countries where P_2O_5 is sold on a water soluble basis.

3. METHODS OF INCORPORATION

The physical forms in which the ammonium phosphates can be produced and so used ~~on a conventional plant~~ for making fertilisers. are as follows:-

(i) in the form of crystals or as a powder produced by grinding either the crystals or, for example, the drum-dried product;
(ii) as a solution or slurry;
(iii) in the form of small granules.

3.1. Use of crystalline or ground MAP

In our first factory-scale tests the aim was to produce a 12:12:18 fertiliser in which all the P_2O_5 was derived from either crystalline or ground MAP. The plants were operated in the normal way, there being a simple replacement of superphosphate by monoammonium phosphate. Ammonium sulphate supplied the extra nitrogen, and potassium chloride (60% K_2O) the potash. The tests were done on both rotary tube and pan type plants. In no case was granulation a practical proposition. Although the amount of water was varied within wide limits and the period in the granulator gradually increased to an impractical time, granules would not form, and the material entering the drier had the appearance of damp sand. This either passed through the drier to give fines with little, if any, product size material, or lodged in the drier shell, where, due to partial solution and then drying, it formed a hard crust which eventually built up and choked the drier. Some improvement was possible by using closely sized ammonium sulphate crystals, but even then the output was far below that required for economic operation. The results of the tests done on the two types of plant were similar.

3.2. The use of a solution or slurry

The well-known Dorr process[2] uses the ammonium phosphate as a hot slurry. This consists of a solution saturated with respect to ammonium sulphate and ammonium phosphate together with both those salts as suspended solids. It is produced by dissolving ammonium sulphate in phosphoric acid and then ammoniating to a chosen pH value. The slurry so formed is mixed with large quantities of recycling fertiliser to which have been added the requisite further quantities of ammonium and potassium salts to give a fertiliser of the desired composition.

This process produces an attractive granular fertiliser, but, owing to the heavy recycles used, normally at least three or four times that encountered on a conventional plant working on superphosphate based fertilisers, the system is not readily applicable. The heavy recycles are beneficial since they reduce the percentage moisture in the drier feed to a point where drying is relatively easy and build-up in the drier does not become a problem.

3.3. The use of small granules

It has been known for a considerable time[3,4] that small granules introduced into a mixture will stimulate granulation. The granules behave as nuclei around which other fertiliser constituents coat themselves to form granules built up like the kernel and shell of a small nut.

Our next tests used small monoammonium phosphate granules ($\frac{1}{2}$ - 2 mm.) as the sole source of water soluble P_2O_5. This technique met with such a large measure of success that the tests warrant description in detail. Fertilisers made in this way included 12:12:18, 15:10:10 and 10:20:6:10*.

3.3.1. Description of plant. The particular plant chosen for this work normally produced superphosphate based fertilisers such as 8:9:13 at about 6 tons per hour, and was equipped with a rotary tube type granulator. In addition there was also a pin granulator[5] which could be used to precede the rotary tube.

The pin granulator is essentially a U-trough mixer in which the rotating shaft carries a series of pins. These are set radially with their tips just clear of the walls of the trough, and are arranged in helical fashion so that rotation of the shaft propels material along the trough. The shaft rotates at fairly high speed, and in addition to propelling material along there is agitation which causes particles to agglomerate. This apparatus had previously been used with considerable success on superphosphate based fertilisers when it had reduced the amount of water required for granulation and had also improved considerably the proportion of product size granules leaving the drier. No great improvement was obtained when the unit was used in the tests using crystalline and ground MAP.

The following modifications were made to the drier, a unit approximately 50 ft. long and $6\frac{1}{2}$ ft. in diameter.

(i) All internal packing was removed from the first 20 ft. of the shell and this was replaced by simple lifters.
(ii) The speed of the exhaust fan was increased so that 15-20% more air was drawn through the drier. The object of this was to reduce the gas temperature entering the drier without reducing the total heat input.
(iii) The feed chute was fitted with a water-cooled jacket to reduce the tendency for damp feed sticking to it.

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*In this formulation the 20 refers to water soluble P_2O_5 and the 6 to water insoluble P_2O_5.

3.3.2. <u>Manufacture of 12:12:18 based on MAP.</u> The fertiliser composition chosen for the first test was 12:12:18 and the plant was started up at a feed rate of approximately 6 tons per hour without the pin granulator in circuit. No granulation occurred in the rotary tube even when excessively large quantities of water were added. It quickly became obvious that the fines recycle was rapidly increasing and the feed rate was reduced to about 4 tons per hour. At this level granulation occurred chiefly in the drier and the plant operated satisfactorily for a further 25 hours. At the end of this period fairly severe build-up had occurred in the drier and it was necessary to remove this before restarting. A summary of the plant operating data is shown in Table I, column 2.

One way to overcome the problem of drier build-up was to reduce the moisture content of the drier feed, and past experience with the pin granulator suggested that this unit would probably meet the requirement. Accordingly, when the plant was restarted, this unit was in circuit and all the water for granulation was added to it. The improvement was immediate and most marked. The feed to the drier which had previously resembled damp sand now was composed almost entirely of discrete granules substantially within the required size range. It became practicable to return to the 6 tons per hour feed rate and the water required for granulation, per unit weight of product, fell to about two-thirds that required in the first test. Typical operating results are shown in Table I, column 3. For comparative purposes in column 4 are shown typical results when the plant was producing superphosphate based fertilisers.

<u>Table I</u>

<u>12:12:18 fertiliser - typical operating results</u>

(1) Item	(2) Without pin granulator	(3) With pin granulator	(4) Superphosphate based 8:9:13
Drier inlet gas temp. °C.	650-750	650-750	450-750
Solids temp. leaving drier °C.	105-120	110-120	105-130
Moisture content of product %	0.2-0.5	0.1-0.3	0.8
Recycle rate - fines (-1½ mm.) tons/hr.	3½-4½	3-4	2-2½
Recycle rate - oversize (+4 mm.) tons/hr.	1½-2½	1½-3	1½-2½
Production rate (1½-4 mm.) tons/hr.	3-4½	4½-6½	6-6½
Total* water for granulation (gal./ton product)	60-80	47-55	33-39

*In the case of the superphosphate based fertiliser, column 4, the moisture content of the superphosphate is included.

It will be seen from a comparison of the columns that the use of the pin granulator had made possible an hourly output equal to that obtained with superphosphate based fertiliser, and 40% higher in terms of plant foods produced.

Fig. 2 is a photograph of the granules produced by this process, and in Fig. 3 the granules have been split to show the ammonium phosphate nucleus surrounded by the shell of the other fertiliser constituents. 80-90% of the granules in the product were formed in this way.

3.3.3. Manufacture of 10:20:6:10 based on MAP. Owing to the high proportion of ammonium phosphate in this fertiliser (35-40%), it was expected that some difficulty might be experienced with granulation. For this reason, and in view of the great improvement obtained when 12:12:18 was made, the pin granulator was used throughout the work on this fertiliser.

Contrary to expectation no difficulty was encountered with the granulation step, and the drier feed consisted almost entirely of well-formed spherical granules of product size, and it was possible to work at a production rate of 6 tons per hour. Some difficulty was at first experienced due to build-up in the drier; this was completely overcome by fitting chains inside the drier along the first 6 feet. These chains were fixed at both ends to the drier shell and hung in catenary fashion so that as the drier rotated they cleaned the shell. As an additional precaution, the lagging was removed from a similar distance along the outside of the drier and additional hammers fitted. As a result of these modifications, no further build-up troubles have been encountered, although well over a thousand tons of ammonium phosphate based fertiliser have subsequently been made experimentally using this plant. Typical operating conditions are given in Table II.

Table II

10:20:6:10 fertiliser - typical operating results

Item	
Drier inlet gas temp. °C.	510-650
Solids temp. leaving drier °C.	95-120
Product moisture %	0.1-0.3
Recycle rate - fines (-1½ mm.) tons/hr.	2½-3
Recycle rate - oversize (+4 mm.) "	1-1½
Production rate (1½-4 mm.) tons/hr.	5-6
Total water for granulation (gal./ton product)	30-40

The advantages to be gained by intimately premixing recycle with the raw materials were particularly noticeable during the granulation of this fertiliser, lack of adequate mixing being shown immediately by the production of either 5-10 mm. granules or of fines. This phenomenon was apparent with other fertilisers but to a less marked extent.

3.3.4. Manufacture of 15:10:10 based on MAP. This composition was chosen as one typical of high nitrogen fertilisers. Considerable difficulty was again expected during the granulation of this fertiliser owing to the large proportion (more than 60%) of ammonium sulphate in the feed.

The plant was again operated with the pin granulator in circuit, and it seemed at first that this fertiliser could be produced as easily as the previous compositions, the discharge from the rotary granulator being quite good although not so uniform as when making 10:20:6:10. After a time it became evident that the granules were breaking down in the drier to produce fines which, when recycled, reduced the quality of the material entering the drier. To produce dry product size material it was necessary to feed the drier with wet 3½-7 mm. granules. Even then it was difficult to produce finished fertiliser at a reasonable rate.

In spite of the employment of a number of expedients, this problem was not solved before it became necessary to end the experimental work on the plant.

4. MANUFACTURE OF FERTILISERS BASED ON DAP

The main disadvantage of using diammonium phosphate in a conventional plant is the loss of nitrogen when the fertiliser is dried. Laboratory tests showed that nitrogen loss could be substantially reduced by coating a DAP nucleus with other fertiliser constituents. Following the encouraging laboratory work, experiments were done on 5 tons per hour plants of both the rotary tube and pan types. The compositions chosen for this work were 12:12:18 and 11:11:11; the latter contained 29% of superphosphate and 10% of DAP of N:P ratio 1.7.

Granulation behaviour was very similar to that experienced when using MAP. The pin granulator again made a marked improvement when used in series on the rotary tube type plant, an output of 5 to 6 tons per hour being achieved with both fertilisers. Without the pin granulator the output from the two types of plant was similar; this was 4½ to 5 tons per hour for 11:11:11 and 3½ to 4½ tons per hour for 12:12:18.

When a mixture of DAP and superphosphate is used to provide the P_2O_5 in a fertiliser, then reversion of part of the water soluble P_2O_5 occurs. The extent of this reversion is in accordance with the theoretical expectation if it is assumed that (a) the DAP decomposes to give MAP and ammonia, (b) the ammonia reacts first with the free acid in the superphosphate, and (c) the rest of the ammonia reacts with monocalcium phosphate to form dicalcium phosphate and MAP.

The loss of ammonia from the 12:12:18 during drying was 0.6 to 2.5% of the total nitrogen present. This loss is less than half that which would be expected if the fertiliser were made by a non-nucleation technique in a conventional plant.

5. REFERENCES

1 RAISTRICK, B. Fertiliser Society Proceedings, No.38. Mar. 22, 1956.

2 DORR-OLIVER INCORPORATED. Brit. Pat. 795,820, May 28, 1958.

3 PHILLIPS, A.B., HICKS, G.C. et al. J. agric. Food Chem., 1958, 6(6), 449.

4 UHDE, F. & PFIRRMANN, T.H. Ger. Pat. 673,216, Mar. 18, 1939.

5 SCOTTISH AGRICULTURAL INDUSTRIES LIMITED. Brit. Pat. Applic. No. 30414/56.
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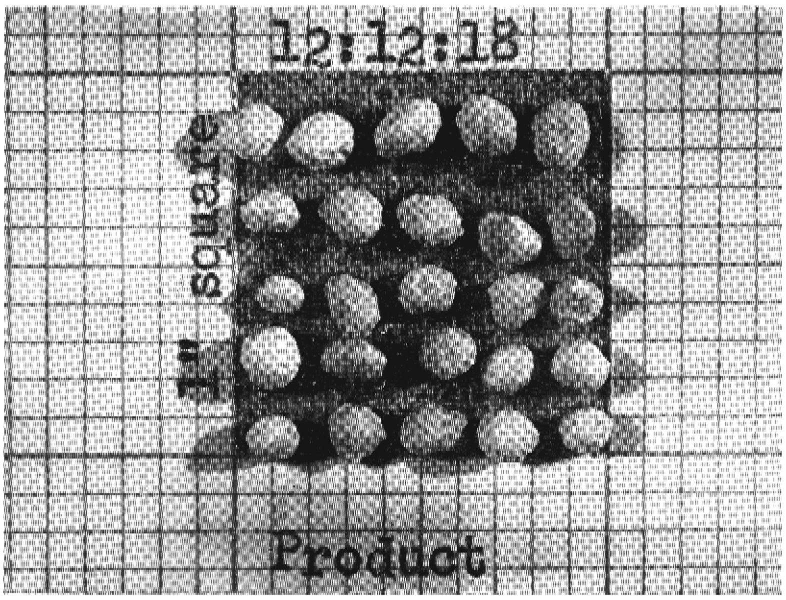

Figure 2

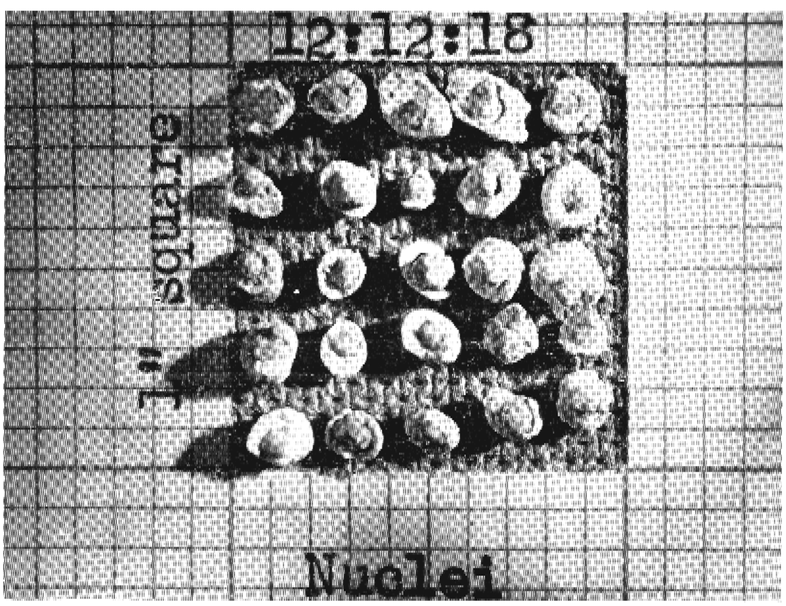

Figure 3

In the post-war years the advantages of high plant food content fertilisers have become increasingly appreciated, and the ammonium phosphates with their 60+ plant food content are being increasingly used. In the past their use in fertilisers was restricted to a few large manufacturers, but today their increasing availability as an alternative raw material to superphosphate makes experiments on their incorporation of wide interest.