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DIRECT PRODUCTION OF GRANULATED SUPERPHOSPHATES AND PK COMPOUNDS FROM SULPHURIC ACID, PHOSPHORIC ACID, ROCK PHOSPHATE AND POTASH

A. Sinte Maartensdijk, Unie van Kunstmestfabrieken BV., Utrecht (Holland)

1. INTRODUCTION

In some countries there has developed a considerable demand for PK compounds, either based on watersoluble Phosphorus or on citric or formic acid soluble Phosphorus. These products are produced in powder or granulated form, usually by mixing matured Superphosphates and Potassium Chloride, for granular products followed by granulation and in most cases by drying to improve storage and handling properties. Other manufacturers simply blend granulated superphosphates with 'granular' potash, a product obtained by a compacting process. The latter method, however, produces a blend which, as a result of the difference in shape and size range of the blended ingredients, may give rise to segregation.

This paper describes a process which deviates from conventional processes as it manufactures superphosphates and/or PK compounds directly from sulphuric and phosphoric acid, rock phosphate and potash without requiring intermediate storage and maturing of superphosphate.

An accurate control of the fineness of rock grinding, constant temperatures and acidulation yield a superphosphate ex Den of a high watersoluble P recovery and low free phosphoric acid content, which can very well be directly granulated.

The advantages of the described process over existing conventional processes for granulated PK compounds are considerable savings in handling and storage, as well as a large increase in capacity for the same size of equipment and resulting into labour and power requirements per unit of product.

2. DESCRIPTION OF THE PROCESS (Fig. 1)

Rock Phosphate is ground to a fineness of 90-98% smaller than 0.1 mm (expect in the case of production of granular triple or single superphosphates) in a roller type mill and fed directly without passing through a ground rock storage hopper on to a weighbelt which transmits a signal to two ratio controllers, i.e. one for sulphuric and one for phosphoric acid. Deviations in Mill output are reduced to a very minimum by carefully controlling the rock feed and air separation system. Variances in the Mill output normally are within 2% over extended periods of time.

Phosphoric acid of a concentration of about 50% P_2O_5 is pumped directly from the storage tanks and measured by means of an electro-magnetic flow meter. Sulphuric acid of a concentration of 96% from the storage tanks is, if required, diluted, cooled and pumped via a small buffertank to the mixer. A very accurate feed rate is controlled also by an E.M. Flowmeter. The slurry, of thoroughly mixed rock and acid, is discharged into a Broadfield superphosphate den.

The gases from the Broadfield den are scrubbed in a high efficiency scrubbing Plant and when larger amounts of sulphuric acid are used, the Fluorine can be recovered as a 20% H_2SiF_6 solution.

The retention time of the superphosphate in the den is maintained at its maximum (35-40 minutes) in order to allow chemical reactions to proceed as far as possible.

If any filler is required to balance the formulation of a product, this is usually introduced into the den, together with the superphosphate slurry.

Superphosphate produced under these process conditions is of a very good physical quality, desintegrates easily into powder by the action of the cutter of the den and does not require any further milling. After leaving the den, it is fed into a bucket elevator, together with a controlled feed of KCl and the recycle from fines screens and cyclone discharge.

Granulation is carried out in a drum granulator at approximately 70°C by steam, sometimes supplemented by water. Moisture content ex granulator is reduced from 10-12% to 4-6% by drying in a rotary dryer. After screening the finished product is cooled in a rotary cooler and, if required, coated, weighed and by means of a belt conveyor system, transferred to bulk storage.

3. FUME AND DUST EXTRACTION IN:

3.1 Rock Grinding Installation

In order to prevent dust formation in the mill building, a certain amount of the circulating air is being extracted from the grinding installation. This keeps the entire unit under a slight vacuum and prevents the escape of any fine dust. At the same time, the extraction of this air (of high humidity), which eventually is replaced by fresh air of low humidity, keeps the circulating air stream well above the dew point so that no condensation of water vapour can occur in cyclones, ducting etc. The extracted air is, after heating up well above the dew point, passed through a bag filter and discharged into the atmosphere.

3.2 Superphosphate Production

The gases from the Broadfield den are passed through a Fluorine Recovery Plant and, when the fluorine concentration is of a sufficiently high level, fluosilicic acid is being recovered as a 15-20% solution. The fluorine recovery unit consists of two scrubbers in series. The first one being a high efficiency scrubber which collects 90-95% of the amount of fluorine as a 15-20% H_2SiF_6 solution. After passing through a knock-out-drum, the remaining fluorine is removed from the gases in the second scrubber which is an ordinary void scrubber; the effluent from this scrubber is not recovered.

Depending on the proportion in which sulphuric and phosphoric acids are used for the production of superphosphate, the concentration of Fluorine in the stack gases varies from 2-25 mg F/nm^3 air (approximately 0.01-0.02 kg F per tons of rock).

3.3 Granulator

Gases from the granulating drum are extracted at in and outlet of the drum and scrubbed with a small amount of water in a separate high efficiency scrubber. Flowmeters are installed on all water supply points.

3.4 Dryer, Cooler and Dust Extraction System

All conveyors inside the granulating plant building and the vibrating screens are enclosed and connected to a central dust extraction system. Cyclone separators are installed to recover dust from the dryer, cooler and dust extraction system and after the three fans, the gases are united into a manifold and passed through a high efficiency scrubber which reduces the fluorine- and HCl-content of the gases down to the desired level before discharge into the atmosphere. Here, too, flow-meters are installed in order to maintain the amount of scrubbing water at the correct level.

4. MATERIALS OF CONSTRUCTION

Because of the corrosive nature of the gases and solids during the production of PK compounds, using potassium chloride, the granulator as well as the first and last part of the dryer are provided with an extra mild steel lining which can be replaced when necessary.

Also, a wide application of plastic material has been made. All ducting of the dryer, cooler, granulator and dust extraction system is made of fibreglass reinforced polypropylene. Also, the cyclones and top parts of dryer, granulator and cooler outfall boxes are made of F.R.P. A temperature alarm system protects, especially the dryer exhaust ducting against damage, due to overheating.

The main scrubber is made of rubberlined mild steel with polypropylene internals (impingement plates, spray pipes and nozzles and liquid separator), whereas the small scrubber for the granulator is made entirely of polypropylene.

5. PRODUCTION RATES, P-RECOVERIES, PRODUCT QUALITY, ETC.

A number of products have succesfully been produced in the Plant, as for instance: 0.10.20; 0.7.30; granular T.S.P.; granular S.S.P.; granular 16% P. Recycle ratios for this process are far less than for the conventional systems where matured superphosphates are used as a raw material.

In table 1 some recycle ratios are given for granulated PK and P Products:

Table 1

Product in % N-P-K	Recycle - Ratio (Recycle : Product)
0- 8- 0	0.63 : 1
0-20- 0	1.25 : 1
0-10-20	0.8 : 1
0- 7-30	0.8 : 1

In the production of 0-10-20 and especially 0-7-30 the amount of oversize during granulation is very small and negligeable as compared to granulation of T.S.P. for instance.

Depending on the degree of acidulation, superphosphates can be produced which have a watersoluble P recovery as high as 94% when leaving the Broadfield den. Some typical examples of production runs are given in Table 2.

Table 2

	Watersoluble P Recovery (%)			Free Phosphoric Acid (% P)			Moisture (%)		
	Ex. Broadf. Den	After granul. and drying	After Maturing	Ex. Broadf. Den	After granul. and drying	After Maturing	Ex. Broadf. Den	After granul. and drying	After Maturing
T.S.P. (20% P)	93	94	98	3.0	2.5	1.65	10-12	5-6	5
S.S.P. (8% P)	88.5-89	89-89.5	92	2.0	1.7	1.4	11-12	5	4.5-5
0-10-20 (N-P-K)	94 -94.5	94	95-96	3.0	1.6	1.3	9-11	5	4 -4.5
0-7-30 (N-P-K)	91	90-90.5	92-93	2.8	0.9	0.6	11-14	4-5	4 -4.5

The screening analysis of finished products is shown in the graph in Fig. 2. On an average, the product contains about 95% of granules between 1.6 and 4.0 mm., 2.4% smaller than 1.6 mm and 2.6% larger than 4.0 mm.

The steam and water requirements for granulation depend to a large extent on the moisture content of the superphosphate leaving the Broadfield den (when keeping retention time and fineness of grinding constant). For process conditions, as described above, the steam and water requirements for the production runs from Table 2 are approximately as shown in Table 3.

Table 3

Product	Steam Cons. kg/t product	Water Cons. kg/t product
T.S.P. (20% P)	50-60	60-65
S.S.P. (8% P)	50-60	70-100
0-10-20 (N-P-K)	10-20	0
0-7-30 (N-P-K)	30-40	0-10

Power consumption and labour requirement for some products are given in Table 4.

Table 4

Product (N-P-K)	Power consumption in Kwh per ton of finished product	Labour required in manh./ton f. prod.
0- 8- 0	33.5	0.208
0-16- 0	38.4	0.250
0-10-20	32.2	0.208
0- 7-30	28.3	0.179

6. MERITS OF THE PROCESS

6.1 Product Quality

Products produced by this process are of a quality which is superior to that of products produced by the conventional methods using solid raw materials followed by granulation or bulk-blending.

6.2 Granulation

The granulation of fresh superphosphates, either straight or mixed with potassium chloride, is much more spontaneous than of matured superphosphates. A very accurate control of rock acid ratios, as well as the rock grinding process is essential, however, when this control is maintained the granulation process is extremely simple and steady.

6.3 Plant capacity

Due to the fact, that granulation is very spontaneous and recycle ratios are only a fraction of those in a conventional Plant, the Plant capacity is, depending on the formulation of a product, about twice the capacity of a conventional unit of similar dimensions.

6.4 Raw Materials

When granulating matured superphosphates mixed with potassium chloride, especially the quality of the latter is very important and it more or less controls the granulation process. Particle size and the addition of anti-caking agents have a considerable influence on granulation and pregrinding is sometimes carried out in order to eliminate or at least to keep this influence within certain limits.

In the described process, different types of potassium chloride have successfully been used without any pregrinding etc. The effect of the different types of potassium chloride on granulation was much less significant than in the conventional process.

6.5 Double Handling and Storage of Raw Materials

As superphosphates are granulated directly from the den and do not have to be stored for maturing in powder form, there is a considerable saving on handling, milling and storage.

6.6 Flexibility

Due to the fact that products are produced directly from sulphuric, phosphoric acid and rock phosphate, the process offers a maximum degree of flexibility.

6.7 Elimination of Acidic Gases and Corrosion of Equipment

The formation of Hydrochloric acid fumes and severe corrosion of equipment is one of the usual features when producing granulated and dried PK compounds. When granulating superphosphates directly ex den the evolution of Hydrochloric acid during granulation, drying and screening is more pronounced than when using matured superphosphate as a raw material. However, when accurate control of rock grinding, acidulation and drying temperatures is exercised, this need not be a problem and the dust and fume extraction system in the Plant does not have to be any more elaborate than in the conventional Plants, in order to maintain good working conditions in the Plant.

By applying suitable materials of construction and a good and practical design of Plant and Equipment generally, corrosion problems, too, are of the same magnitude as for conventional units of production.

6.8.1 Power Consumption

As shown in Table 5, electrical power consumption is somewhat lower for the production of PK compounds, and granulated superphosphates by avoiding extra handling and storage.

Table 5

Product (N-P-K)	Electrical Power Consumption in Kwh per ton of Product				
	Conventional Process				Direct gran. of Superphosphate
	Superphosphate production	Milling of Superphosphate	Granulation	Total	
0- 8- 0	12.9	2.5	24.2	39.6	33.5
0-16- 0	12.9	2.5	29.0	44.4	38.4
0-10-20	7.7	1.5	24.2	33.4	32.2
0- 7-30	5.2	1.0	24.2	30.4	28.3

6.8.2 Labour Requirement

In Table 6, a comparison is made between labour requirements for conventional methods of production and for the direct granulation.

Table 6

Product (N)	Labour required in manhour per ton of finished product				
	Conventional Process				Direct gran. of Superphosphate
	Superphosphate production	Superphosphate milling	Granulation	Total	
0- 8- 0	0.100	0.067	0.438	0.605	0.208
0-16- 0	0.100	0.067	0.525	0.692	0.250
0-10-20	0.060	0.040	0.438	0.538	0.208
0- 7-30	0.040	0.027	0.438	0.505	0.179

6.9 Future Developments

The Plant has been in operation since 1974 and has successfully produced a number of different grades of superphosphates and PK compounds. Further possibilities are NPK compounds which can be produced in the same Plant by the addition of a nitrogen carrying raw material. For instance, granulated 5-5-10 (N-P-K) has successfully been produced directly from sulphuric acid, rock phosphate, potassium chloride and ammonium sulphate.

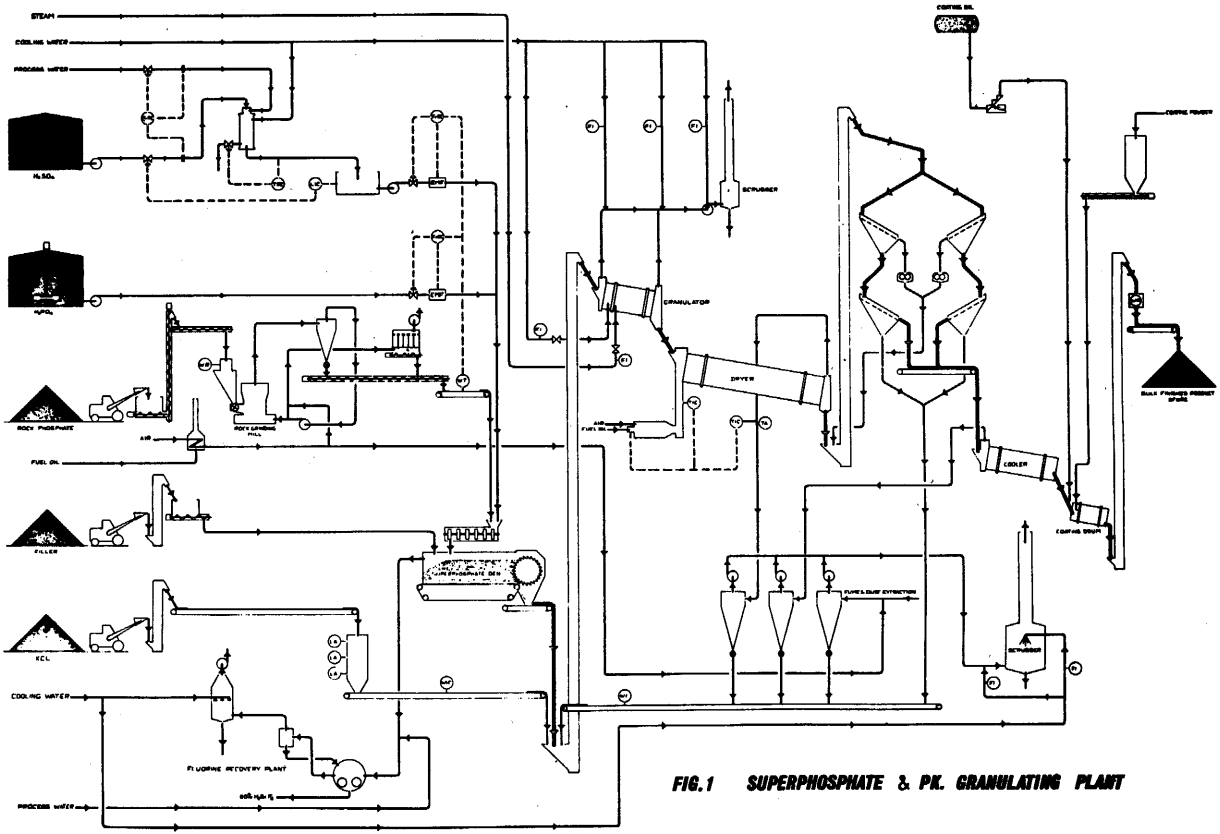

FIG. 1 SUPERPHOSPHATE & PK. GRANULATING PLANT

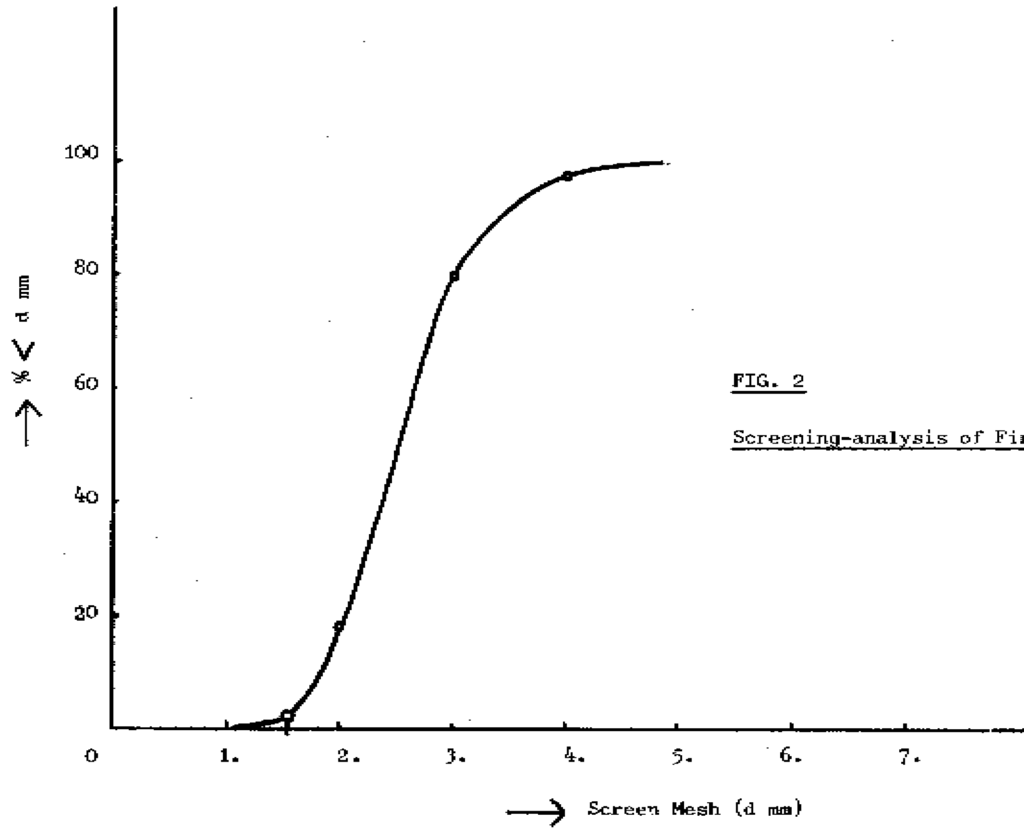

FIG. 2

Screening-analysis of Finished Product