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PRODUCTION OF NPK FERTILIZERS WITH A SPOUTED BED GRANULATOR - DRYER

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SUMMARY

Agrimont set up a technology producing NPK fertilizers based on granulation - drying of ammoniated slurry from nitric attack of phosphate or sulphuric-phosphoric acid mixture carrying out a research work on a micro-pilot and pilot plant.

The commercial plant based on said technology has been operating for 5 years in the Priolo factory (Sicily), producing DAP, NP fertilizers by nitric attack of phosphate rock (15-30-0), NPK fertilizers with potassium sulphate (20-10-10, 12-24-12, 15-15-12) and with potassium chloride (15-15-15).

The production capacity of the Priolo plant, based on a single spouted bed, ranges from 500 to 700 t/d according to the product grade and water balance.

The performances obtained with the spouted bed process are compared with the drum granulation process here below.

The main features of the spouted bed process are the simple flowsheet involving lower investment and production costs, ease to run and control by automatic devices.

The total investment cost for a plant based on the spouted bed process and with a capacity of 180,000 to 200,000 tons/year of NPK fertilizers is estimated at US \$10.5 million (about 20% less than an equivalent plant based on the rotary drum process).

The yearly economic advantage for the spouted bed process is about US \$ 900,000, mainly due to the lower investment and direct costs.

INTRODUCTION

The history of fertilizer granulation follows the history of chemical tech nology. Superphosphate was first granulated in 1921 and was the first fertilizer that underwent agglomeration for producing round granules. Since then, the granulation process has had a steadily increasing development which was surely the reason of the success of fertilization in the period of agricultural mechanization.

Numerous authors have written a lot on the granulation phenomena and it is not the task of this report to speak about the arguments and problems that were already solved a good while ago (1) (2) (3) (4).

We would just like to recall, among the various granulation processes operating in industrial scale, the most popular ones employed. These are the ones based on the agglomeration, in which the phenomenon of the volume increasing of the granules is carried out by adhesion of primary fine particles utilizing a liquid bond coming from the so-called "liquid phase" and those where the granules were enlarged by means of adding layers of solution or slurry according to the "onion skinning" model.

Another difference among the various processes to be mentioned is the type of drying :

- The production processes where the drum granulator is followed by a drum dryer in which the granules harden due to the formation of bonds of various nature owing to the evaporation of the water from the liquid phase.
- The processes where the granulation and drying operations occur at the same time. Spouted bed granulation is situated among these kinds of processes where the "onion skinning" model is applied.

Agrimont was first in this matter in the sixties aiming at only producing DAP, first of all with a pilot plant, and then with an industrial plant having a capacity of 250 t/d DAP in the Priolo factory. Studies followed on into the seventies with the aim of extending the employment of the spouted bed technique for producing NPK fertilizers in general, and for nitric attack in particular, which seemed the most suitable to be granulated using this technique.

With this aim in mind, a pre-pilot plant having a capacity of 20-30 Kg/h was built in the Porto Marghera Fertilizer Research Center. The chemical and chemical -physical behaviour of the various slurries and the parameters of the process that control the rheological behaviour of the slurries as well as the parameters that regulate the granulation were studied and defined.

A pilot plant having a 1,500 - 1,800 Kg/h capacity was also built at Priolo, where the geometric and fluid dynamics were studied and developed so as to optimize the granulation conditions for various formulas and to obtain scale-up data for the design of an industrial granulator.

The new industrial granulation apparatus having a 500-700 t/d capacity was installed in an existing granulation plant, appropriately modifying the cycle and adding a new scrubbing section.

The plant was modified in 1980 and has been running with the new cycle and achieved the performances indicated in following paragraphs.

THE SPOUTED BED GRANULATION PROCESS

Apparatus description

Granulation occurs in the apparatus shown in Figure 1; it is made up of a conical shaped section (A), situated under a cylindrical section (B) which is connected to a second cylindrical section (C) functioning as an expansion chamber. On the wall of the cylindrical part (B) and the conical connection

to the cylindrical section (C) there is an outlet for the discharge of the granules (D) which can be regulated at various levels.

The throat of the lower cone is connected to a pipe through which a stream of hot air is fed (350 - 500°C) at high velocity. The air, which is delivered through a blower, is pre-heated in an oven using fuel oil or natural gas.

There is a sprayer in the center of the throat (and therefore in the center of the hot air stream) to atomize the slurry to be granulated.

The granulation process occurs according to the dynamics shown in Figure 2.

The hot air stream, penetrating the bed contained in the conical part of the granulator, produces a fountain of granules thus creating an elevated circulation of material. The bed is made—of crushed oversize and under size granules returning from the screening section.

Whenever the granules are taken into the air jet, they come into contact with the droplets created by the sprayer and thus are covered with a layer of slurry. In the rising stage, part of the water contained in the slurry evaporates rapidly thus cooling the air and the granules are covered with a thin layer of wet solid material.

When the granules reach the top of the fountain, they fall back to the spouted bed and are pulled down towards the hot air stream. In this area, which is kept expanded (but not fluidized) by the hot air which passes through it, the granules undergo final drying.

After staying in the granulator for a period of time depending on the granulator hold-up, on the hourly capacity and the number of external recycling, the granules are discharged from the granulator.

2. Granulation parameters

In sported bed granulator, granulation occurs mainly through the coating of pre-formed granular seeds with successive layers of slurry according to the "onion-skinning" model. The new layers are immediately dried by the fluidizing air at high temperature and velocity. The granules thus obtained are therefore very regular and almost round.

Indeed, the possible irregularities on the surface of the granule are absorbed by the following film of slurry; this can be considered as a nemedial action due to the granules being rubbed together and which file down the possible surface roughness.

The factors affecting granulation can be divided into two groups.

The first group is connected to the chemical and chemical-physical characteristics of the slurry. The following parameters can be observed:

- water content in the slurry
- slurry viscosity

- content of highly soluble salts
- nature of suspended solids in the slurry

Slurry having low viscosity is the most indicated for granulation. The research work carried out in the pilot plant at our Research Center had the aim of obtaining low viscosity slurry with minimum water content and utilizing various raw materials in order to favor both granulation efficiency and production capacity.

Taking into account that NPK slurries are easily non-Newtonian (pseudo plastic or thixotropic) an important laboratory study has been carried out in order to obtain. Newtonian slurry with a minimum water content; as already known, a fluid is Newtonian when its viscosity does not change as a function of shear stress.

Studies were conducted on partially and totally ammoniated slurries, and on slurries containing dissolved potassium salts.

Through these studies, the characteristics of the agitators in the ammoniation and salt dissolving tanks were defined.

Figure 3 represents the viscosity vs.their water content for final slurries of product grades 18-46-0, 15-15-15, 15-15-12S and 20.10.10S.

As regards the solubility of the salts, the granulation is favored by ammonium nitrate, which is always present in nitric attack slurries as long as its content does not exceed 60% in the products containing potassium as sulfate and 50% in the products containing potassium as chloride.

Regarding the influence of the suspended solids in the slurries, it has been shown to be important that almost all the calcium during the attack phase precipitates in the S products, so as to have it in the final slurries in the form of hemi-hydrate sulphate or complexed as $K_2SO_A \cdot SCaSO_A \cdot H_2O$.

This occurs by feeding all or part of the potassium sulphate and eventually ammonium sulphate during the attack phase until reaching a precipitation equivalent ratio (moli $SO_A/moli$ CaO) approximately 1.

As regards the effect of the impurities brought by the phosphate rock, it is to be noted that iron, magnesium and aluminum do not cause any inconvenience in the granulation; on the contrary, they help in those formulas in which there is a low quantity of ammonium nitrate.

But, whenever these metals are present in high quantities (over 3.5% Fe_2O_3 + Al_2O_3 in the phosphate rock) drying problems may arise.

The second group of parameters which affect the granulation is connected to the characteristics of the granulation apparatus; the main factors are:

- the ratio between the hold-up in the granulator and the hourly flow of feed slurry. The lower this ratio, the bigger the granules and vice versa.

- The distance of the sprayer from the granulator throat. If the distance is excessive, it reduces the chances of the granules contacting the slurry droplets which dry, thus causing the formation of microgranules and dust.
- The temperature of the spout air. It must be optimized according to the case. Generally an increase in the temperature favors the granulation of slurries poor in ammonium nitrate. However this can have an adverse effect causing the decomposition of the di-ammonium phosphate.
- The external recycle ratio. This does not have very much effect considering the high internal recycling. In some cases an increase in the recycling ratio (which is usually around 1) up to 2 can solve cases of anomalous gramulation. This operation can be carried out by recycling a certain amount of the final product.
- The spray fluid. A mixture of air and steam is employed for the micronization of the slurry.

THE PRIOLO INDUSTRIAL PLANT

1. Process and plant description (Figure 4)

The plant can be divided into three section: slurry preparation, granulation and salt cycle, and the effluent scrubbers. The slurry preparation section applies the traditional flowsheet defined as sulphuric-phosphoric-nitric attack of the phosphate rocks. The attack of the phosphate rock is carried out through two agitated reactors and is followed by two traditional ammoniation reactors.

The potassium salts are dissolved in the ammoniated slurry in a specific reactor with agitator. This slurry is then fed to the granulation section by centrifugal pump.

The granulator-dryer, working as already described, produces a granulate having a high percentage of good product, which is fed to the screening section; this is then cooled in a drum, treated with anti-caking and sent on to the storehouse.

The screen oversize granules are ground and then recycled to the granulation section together with the fine.

The drying gas, produced in an oven fueled with natural gas, is sent to the granulator in the range of 350° - 500°C depending on the type of product to be granulated. The gases leaving the granulator-dryer are first of all sent to dry cyclone separators, then washed with partially ammoniated phosphoric acid and last of all washed with water.

2. Type of products and operating parameters

The main products obtained industrially are :

18 - 46 - 0

15 - 30 - 0

12 - 24 - 125

15 - 15 - 15

15 - 15 - 125

20 - 10 - 108

Excellent conditions have been found for each product, regarding both the preparation of the ammoniated slurry as well as the granulation stage.

The attached Table 1 shows some typical formulations, the standard daily capacity for each product grade and the consumption of raw materals and utilities per ton of product.

The operating conditions of the plant depend on the type of product to be granulated; however, it can be confirmed that for product grades of the same group, these conditions fluctuate within strict limits.

The attached Tables 2 and 3 reports the most important operating parameters and the main characteristics for some of the products mentioned.

3. Process Control

Plant performance is monitored by automatic instruments which provide accurate measurements of specified variables. A description of the main controls carried out and equipment used is outlined below.

3.1. Chemical controls

The computerized analytical system developed by Agrimont Fertilizer Center allows a real time control of the process slurry by completely automatic sequences. By the control of the different sections of the plant, it is possible to mantain the concentration level of slurries at a steady state.

The result is a good constant grade of the products obtained. A block diagram of the apparatus is shown in Figure 7.

Briefly, the computing system controls the different analytical steps consisting of exact weighing of sample, dissolution of fertilizer granules, filtration and transfer of analytical solution into analytical system, computation of signals from amplifier, printing of results as percentage N-NH3, N-NO3, K_2O , and P_2O_5 , printing of raw materials to be fed in order to maintain the desired concentration level of slurries.

The analytical system consists of a four channel thermometric analyzer. A peristaltic pump drives sample solutions and reagent streams through glass coils in a temperature controlled water bath, so that both streams reach the same temperature. Both streams finally mix inside a thermometric stirred cell. The enthalpy variation given by specific reaction are in linear relationship with the concentrations in the sample.

3.2. Chemical-physical controls

The granulation loop is controlled by monitoring the acidity and viscosity

of the slurry to be granulated.

Thus the spouted bed plant in Priolo is equipped with process pH meters and a process viscosimeter. The pH meter, which can work at high temperature, is equipped with a self cleaned antimony electrode, which responds in temperature compensation.

The process viscosimeter is the coaxial cylinder rotating type. The apparatus measures the required strength to maintain the rotor at constant rotational speed.

The granulation loop of the spouted bed is controlled by continuously measuring the temperature and the moisture of the product leaving the granulator. A standard thermocouple is used for measuring the temperature.

The moisture meter, installed at a given distance above the continuous flow of granules, compares the infra-red energy impinging the granules and the energy reflected by the granules. The quantity of energy absorbed is proportional to the water content in the product.

4. Running and maintenance data

The plant is normally run by 3 shift workers; there is little work to be done seeing that the plant is well automated.

Having little hold-up, the plant is easy to start up.

Also the stops for changing formulas require only a little time; a normal formula change without plant cleaning requires 8-10 hours from the beginning of the stop to the steady running of the new formula. Breaks for cleaning operations depend on the product grade: these breaks are done every 12 (20-10-10) to 20 days (18-46).

The granulator has an automatic washing system which takes a total time of 10 to 20 hours. The plant requires little maintenance due to the low number of apparatures.

COMPARISON WITH A PLANT BASED ON DRUM GRANULATION

1. Process Flowsheets

The optimized flowsheet of a plant based on nitric attack of the phosphates and spouted bed granulation is shown on the attached Figure 5. The substantial difference regarding the plant operating in Priolo is the addition of a high efficiency ammoniation of the slurry and a system of scrubbing the effluents leaving the dryer-granulator based on high efficiency scrubbers.

The drum granulation flowsheet in comparison is shown in Figure 6. This refers to a cycle based on nitric attack of phosphate rock with high efficiency ammoniation and drum granulation according to the traditional flowsheet.

2. Energy Consumptions

The comparison of the specific consumptions and utilities costs relative to the two processes for some typical products of the two processes is shown on Table 4. The raw materials have not been compared seeing that the formulas are the same and the usual yields in the two cases are the same. It can be seen that the spouted bed process requires more electric energy but less fuel oil and steam.

3. Operating and maintenance costs

Operating labor

The spouted bed granulation plant requires three shift workers (1 panel controller,1 worker in charge of the salt cycle and effluents (including granulator), 1 in charge of the reactors.

The drum granulation plant requires 4 shift workers (1 panel controller, 1 granulation worker, 1 in charge of the reactors and 1 in charge of the salts cycle and effluents).

Maintenance

Maintenance costs are lower—for the spouted bed plant because of the lower number of apparatuses, lower recycling ratio in granulation and only one stationary apparatus (spouted bed) as compared to two separate apparatuses in movement (drum granulator and drum dryer).

The same conclusion can be reached considering that the maintenance cost is a fixed percentage of the investment cost.

4. Investment cost

The cost of battery limits investment for a drum granulation plant having a 180,000 - 200,000 t/y capacity is estimated at U.S. \$ 13.2 million whereas the spouted bed granulation plant is estimated at U.S. \$ 10.5 million.

CONCLUSION

From the analyses carried out (see Table 4) it can be found that the process based on spouted bed granulation is more advantageous in respect to the process based on drum granulation.

Main features of the process are :

- lower investment costs
- lower energy costs
- lower manpower and maintenance costs
- easier control due to automated systems
- the excellent product properties, in particular the low water content, the uniform size, the high crushing strength, the excellent storage and handling properties.

Process limitations are :

- the slurries must be pumpable; for this reason, when the slurry contains salts with low solubility, it is necessary to dilute it, thus losing some of the energy benefits;
- the slurries have to be completely ammoniated before granulation; this factor makes ammoniation more critical in respect to the drum granulation, where it is possible to carry out ammoniation of the product in solid form; however, only a good ammonia scrubber unit is required in order to compensate this disadvantage.

Thus it can be definitely concluded that the spouted bed process is very suitable for nitric attack products having a N/P_2O_5 ratio in the range from 2/1 to 1/1.

For products with high P_2O_5 (MAP, DAP), the process appears less advantageous because of having to work with diluted slurries at the maximum degree of ammoniation (DAP). In this latter case, the process appears suitable for little plants where there is the advantage of low investment and where the higher energy cost has a lower influence.

The spouted bed process is also advantageous when diluted phosphoric acid is available: in this case the concentration of the phosphoric acid is not necessary, because the energy efficiency in the spouted bed dryer is comparable to the energy efficiency of the concentration with steam.

REFERENCES

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TABLE 1 - PRIOLO SPOUTED BED PLANT : RAW MATERIALS AND UTILITIES CONSUMPTION (per ton of product)

MATERIAL OR UTILITY		Product grade					
		15-15-12S	151515	18-46-0	20-10-105		
32% P ₂ 0 ₅ Phosphate rock	Kg	183.8	192.3	-	128.8		
56% Nitric Acid (as 100%)	Kg	309.6	316.4	-	459.2		
90-98% Sulphuric acid (as 100%)	Kg	20.1	_	56	-		
40% P ₂ 0 ₅ Phosphoric acid (as P ₂ 0 ₅)	Kg	95.8	93	474	62		
Gaseous ammonia	Kg	104.1	102	225.5	126		
50.5% K ₂ 0 Potassium sulphate	Kg	240	_	-	200		
60.5% K ₂ 0 Potassium chloride	Kg	-	250.4	-	-		
Coating agent	Kg	5	5	-	5		
Electric power	KWh	50	60	68	62		
L.P. Steam	Kg	45	50	60	55		
Fuel oil or gas	Kcal	120,000	130,000	170,000	150,000		
Make-up water	m 3	0.03	0.03	0.04	0.04		
Plant daily capacity	t/d	700	600	500	550		

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TABLE 2 - PRIOLO SPOUTED BED PLANT - MAIN OPERATING PARAMETERS

			4 - 4			
		Product grade				
		15–15–128	15–15–15	18-46-0	20-10-108	
Flowrate of final product	t/hr	29	25	21	23	
Rheological parameters of the sl to the spouted bed granulator	lurry					
- viscosity at 80°C	срв	15–20	15–20	25–30	13-16	
- density at 50°C	Kg/m ³	1,650	1,600	1,480	1,610	
- ammoniation grade		1.55 -1.6	1.5-1.6	1.8-1.9	1.6-1.7	
- water content	wt%	14-17	14-17	20–25	14–17	
- temperature	•c	90-100	80–100	90–100	100-110	
Granulation parameters						
- spouting air flowrate	Nm ³ /hr	33,500	34,500	32,500	35,500	
- spouting air temperature	•c	440	410	480	380	
- air outlet temperature	•c	105	103	85	105	
- bed pressure drop	bar	0.25	0.26	0.23	0.25	
- product temp. at bed exit	•C	105	103	85	105	
					<u></u>	

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TABLE 3 - PRIOLO SPOUTED BED PLANT: FINAL PRODUCT TYPICAL CHARACTERISTICS

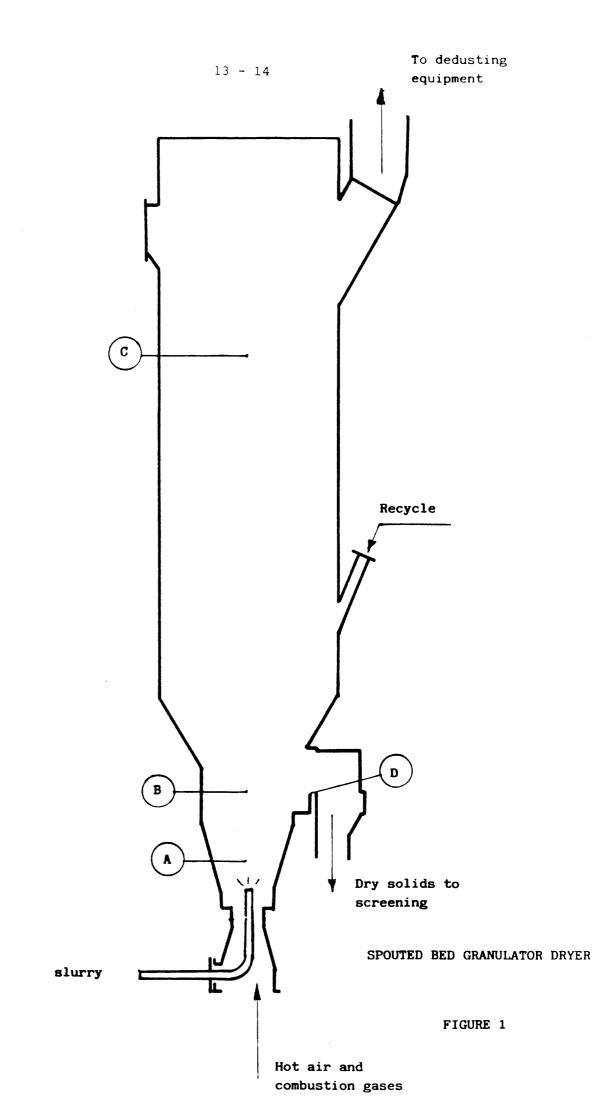
12S 15-15-15 x 2 max	5 18-46-0 2 max	20-10-10S
	2 max	
		2 max
25 22 – 26		15 - 20
70 58 - 63	57 - 62	70 – 75
10 10 – 14	10 - 14	8 - 10
ax 0.2 max	0.2 max	0.2 max
6 5 - 6	4 - 5	5 - 6
0.6 - 0.7	1.2 - 1.5	0.5 - 0.6
5.9 5.2 - 5.3	6.7 - 6.8	5.3 - 5.5
(ax 0.2 max 6 5 - 6 0.8 0.6 - 0.7	ax 0.2 max 6 5 - 6 0.8 0.6 - 0.7 1.2 - 1.5

^{*} All products are nearly spherical, free from dust and free flowing ever after long storage in bulk.

TABLE 4 - ECONOMIC COMPARISON OF SPOUTED BED GRANULATION VS. DRUM GRANULATION (U.S. \$)

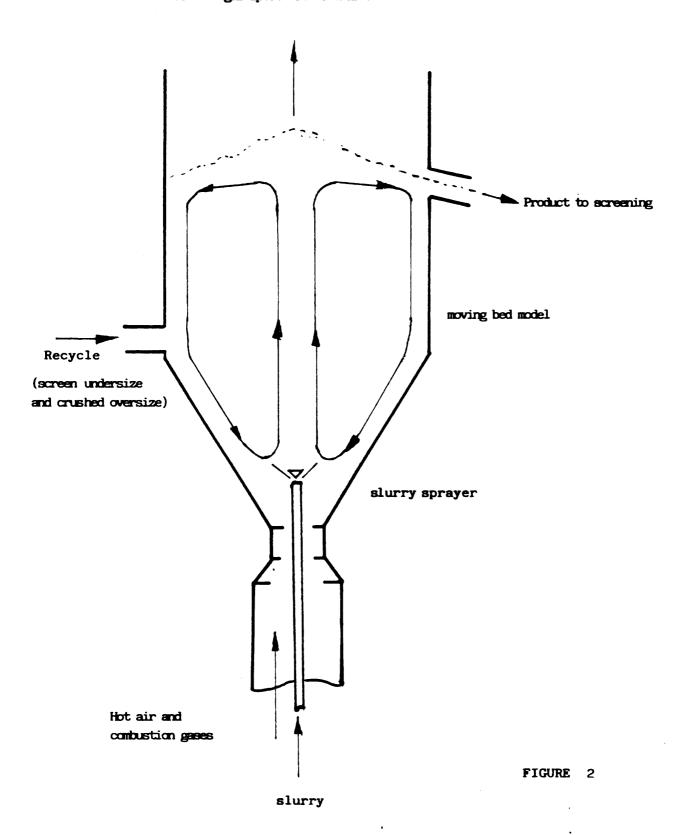
	Spouted bed granulation				Drum granulation			
Product grade	15–15–15		15-15-12S		15–15–15		15–15–12S	
Variable cost	Unit/ton	US \$/t						
Raw materials		a		b		a		b
Utilities								
electric power (0.05 \$/KWh)	60	3.00	50	2.50	35	1.75	35	1.75
fuel gas (0.12 \$/10,000 Kcal)	135,000	1.62	123,000	1.48	150,000	1.80	150,000	1.80
steam (0.01 \$ /Kg)	50	0.50	50	0.50	100	1.00	100	1.00
Total variable cost		5.12 + a		4.48 + b		4.55 + a		4.55 + b
Yearly capacity	180,000		200,000		200,000		200,000	
Yearly variable cost	921,600 + (a x 180,000)		896,000 + (a x 290,000)		910,000 + (a x 200,000)		910,000 + (a x 200,000)	
Operating labor (100,000 \$/shift)	300,000		300,000		400,000		400,000	
Yearly maintenance (5% investment cost)	525,000		525,000		660,000		660,000	
Return on investment (25%)	2,625,000		2,625,000		3,300,000		3,300,000	
Total cost (\$ / year)	4,371,600+ (a x 180,000)		4,346,000+ (b x 200,000)		5,270,000+ (a x 200,000)		5,270,000+ (b x 200,000)	
Differences	+ 25,600		_		+ 924,000		+ 924,000	

REMARK: a and b are the cost (\$/t) of raw materials, considered equal, for both product grades.



OPERATING PRINCIPLE OF A "SPOUTED BED" GRANULATOR

Central gas spout with entrained



VISCOSITY OF FINAL SLURRIES TO SPOUTED BED GRANULATOR

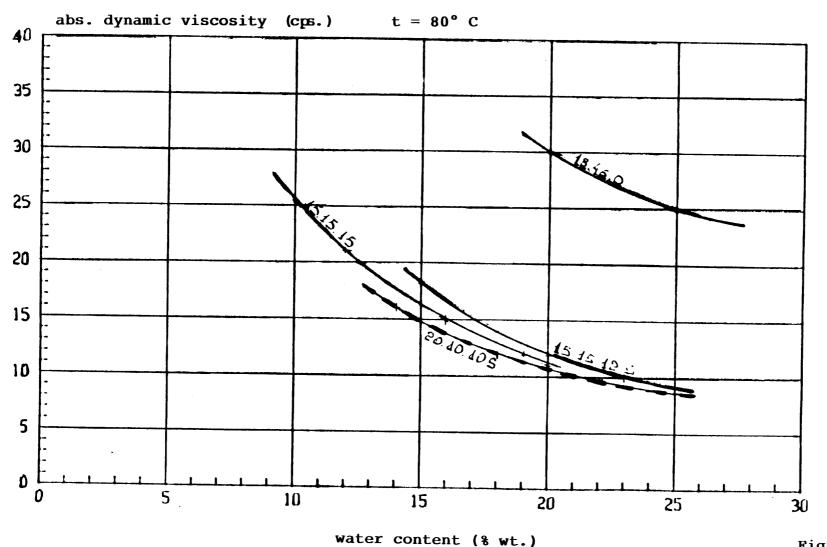
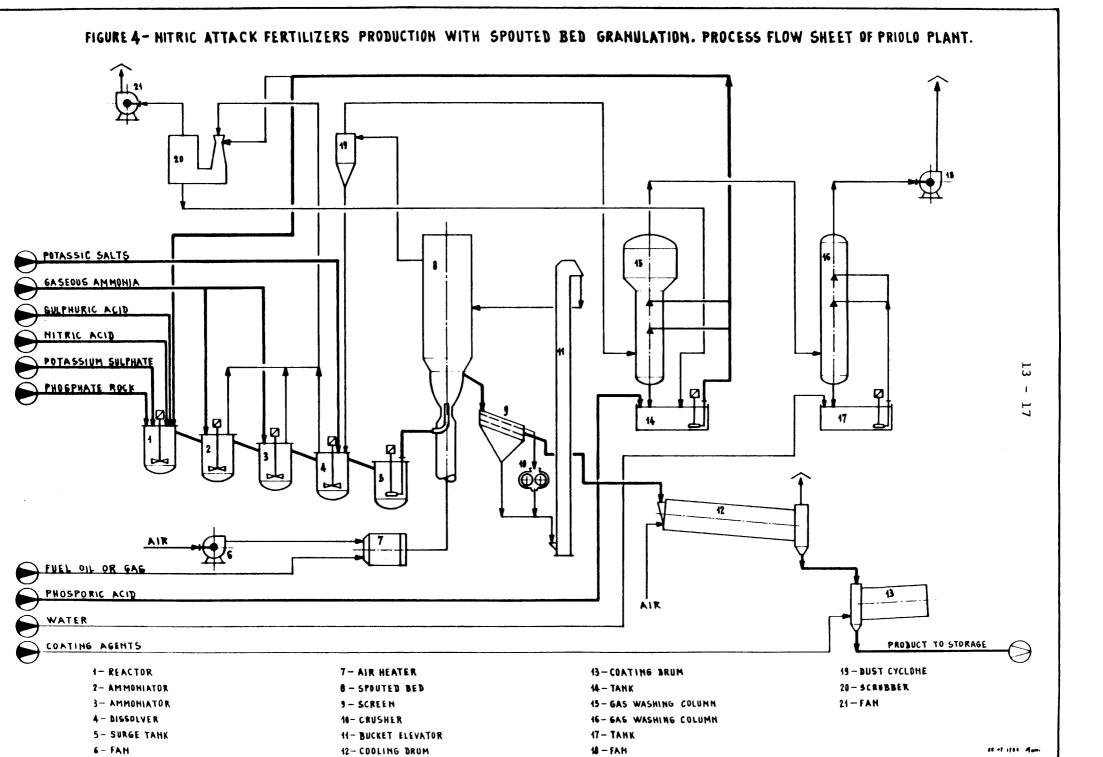
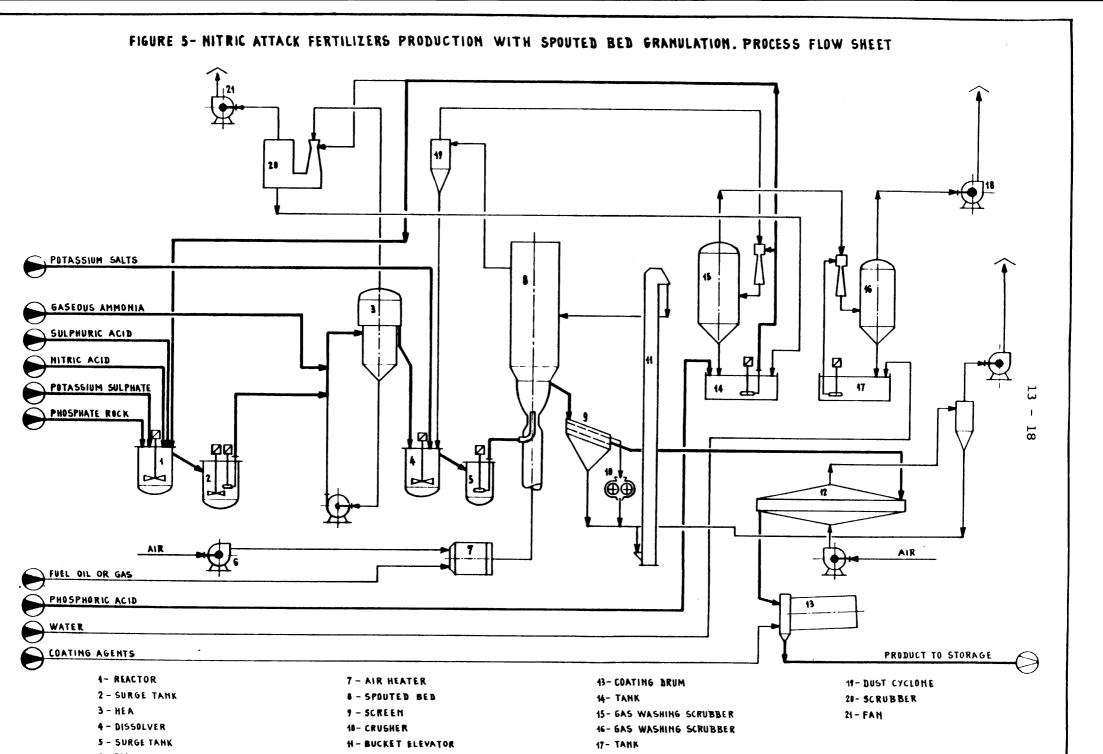


Figure 3





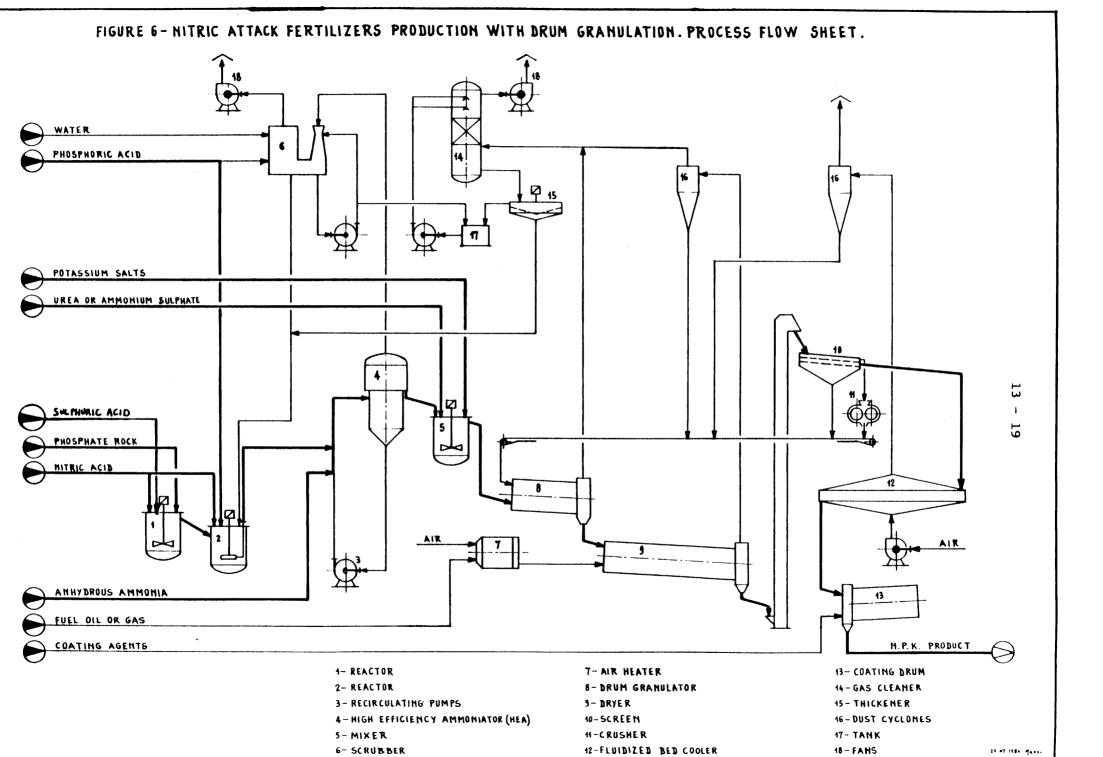
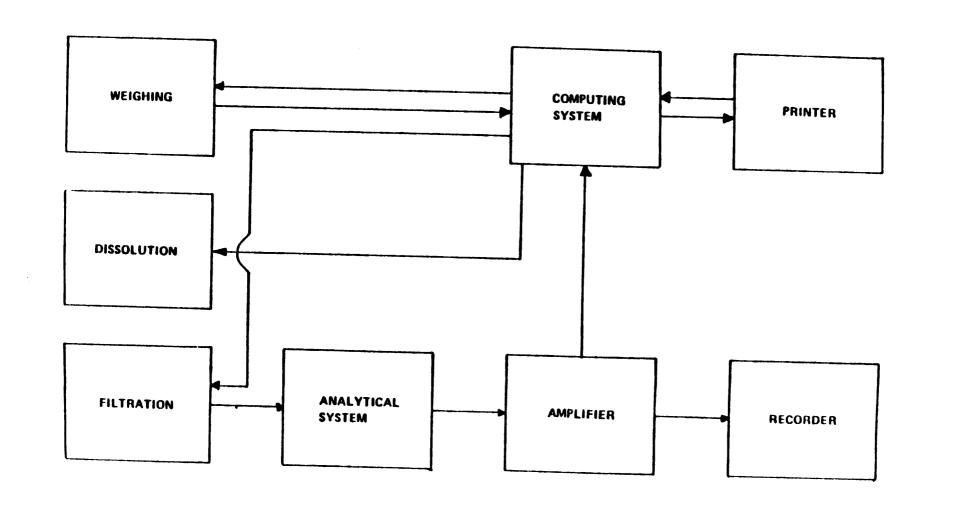


FIGURE 7 . Block diagram of apparatus



TA/86/13 Production of NPK fertilizers with a spouted oed granulator-dryer by G. Brusasco, R. Monaldi, V. de Lucia & A. Barbera, Agrimont SpA, Italy

DISCUSSION: (Rapporteur B. Christensen, Superfos, Denmark)

- W Mr. P. SUPPANEN, Kemira Oy, Finland
 - 1. The inlet temperature of drying gas is high. Does it affect the melting of granules?
 - 2. The temperature of slurry is low and the moisture content is nigh. What are the reasons for these figures?
 - 3. The moisture meter is working with infra-red light. What is the accuracy of this meter? Does it work reliably with all formulations?
 - 4. What is the effect of high efficiency ammoniation (HEA) compared with the old mixing reactor type?
- A 1. No, the slurry is subjected to a quenching phenomenon and solidifies on the granule surface, that grows according to the onion skin model; the temperature is the same in all parts of the bed.
 - 2. The moisture content is connected with the slurry viscosity and the temperature is about at the boiling point of the slurry.
 - 3. The moisture meter is very accurate for all the formulations; the accuracy is \pm 3%.
 - 4. The main advantages of High Efficiency Ammoniator on the mixing reactors are:
 - nigher efficiency of ammonia absorption
 - possibility to reach easily high degree of ammoniation
 - lower investment and operating cost
 - lower investment and operating cost for the abatement equipment, because the evolved steam contains less ammonia and air.
- Q Mr. T. LAINTO, Kemira Cy, Finland
 - I have understood that you have an automatized analyzing system in your plant for nutrient control.

What nutrients (N-NH4, N-NO3, P205 etc...) are you analyzing?

Is the sample taken from granulated material or from slurry?

How is the sample taken and handled before analyzing it?

A - The analyzed nutrients are N-NH4, N-NO3, P2O5 and K2O and the results given by the system are in agreement with official EEC methods.

The system can work either on the slurry or on granulated product.

Samples are fed to the analyzing system by an automatic device, working on line.

- Q Mr. S. ORMBERG, Norsk Hydro, Norway
 - 1. What experience do you have with straight nitrogen fertilizers like AN, CAN and urea?
 - Will the bi-uret content of urea increase?
 - 2. For forest fertilizer bigger granules are preferred. Can your process economically make for instance granules of nitrogen fertilizer of 10 mm diametre?
- A 1. We have no industrial experience of AN, CAN and urea; on these products we have only experience on pilot plant scale.
 - The forest grade fertilizer is not requested by our market; therefore, we have not studied the production of large granules.
- Q Dr. V. SCHUMACHER, BASF, Germany

You told that the energy-consumption is less for the spouted-bed-reactor. In your table 4 energy-consumption is 4.55 \$/t for the drum granulation and 5.12 \$/t for the spouted-bed granulation. Can you explain the difference?

A - I confirm that in the considered cases the energy consumption is less for the spouted bed reactors, when the production capacity of the plant is the same.

Table 4 reports 4.55 US\$/t from drum granulation and 5.12 US\$/t for spouted bed granulation, while the production capacities are respectively 200.000 t/y and 180.000 t/y.

When both production capacities are 200,000 t/y the energy consumptions are 4.48~US\$/t for spouted bed granulation and 4.55~US\$/t for drum granulation.

Q - Mr. Y. LOUIZI, SIAPE, Tunisia

In the NPK spouted bed process, what is the optimum linear velocity of the not air? The heat transfer coefficient? Are there material entrainment by air?

A - The hot gas velocity in the throat is 60-80 m/s.

The heat transfer coefficient is very high.

The dust entrainment from the spouted bed is 3 to 10% of the production according to the product formula, but 95% of the dust is recovered by the dry cyclones.