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A NEW PROCESS FOR THE PRODUCTION OF
CONCENTRATION AMMONIUM NITRATE SOLUTIONS

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(The fertilizer process, recently brought by CdF CHIMIE AZF to its subsidiary GRANDE PAROISSE, have kept their brand name AZF ; that is why AZF only will be used in this paper).

ABSTRACT

The process used to produce ammonium nitrate solutions from nitric acid and ammonia have not much changed during the last decades. Practically all the production in the world is obtained using two types of process implying a large reaction mass :

- atmospheric pressure process which require a relatively simple equipment.
- pressure process : their major advantage is a proper utilization of the neutralization heat, which is particularly advantageous in old plants, where only dilute nitric acid is available. Their main disadvantage lies in the high investment cost, which results from the comparatively complex flow diagram, needing many stainless steel vessels.

AZF has developed a new process using a pipe reactor of a design fairly similar to that of the AZF Pipe Reactor producing ammonium phosphates.

It is particularly advantageous in that it :

- needs very simple equipment, mainly consisting of a pipe reactor and a separator, and, if necessary, a mist scrubbing system,
- provides a high efficiency (above 99 %)
- warrants a high operational safety, since the reaction mass is minimized.
- offers the possibility of producing directly a nitrate solution at a concentration of 97 % when using 63 % nitric acid as basic material.

INTRODUCTION

The process used worldwide to produce all the ammonium nitrate solutions are very similar and have not much changed during the past decades.

It would be too long to review here the 15 or maybe 20 process, which have been developed in the course of the years, to manufacture ammonium nitrate solutions. Interesting studies on this subject are in the books published by HONTI (1), SAUCHELLI (2) and KELETI (3).

The general design of all these process take care of two major concerns :

- avoid the formation of ammonium nitrate aerosols, which tend to appear easily, for the boiling point of the nitric acid is lower than the temperature reached in the reaction mixture.
- limit the risks of decomposition of the ammonium nitrate.

A third concern has appeared in the most recent process, which also are more complicated : utilise most of the important heat of reaction available, to produce a solution as concentrated as possible.

The various process fall into two categories, according to the pressure of reaction : non-pressure process and pressure process :

1/ Non-pressure process are simpler than pressure process.

Some process of the first category are even rudimentary, without any energy recovery, and have poor yields.

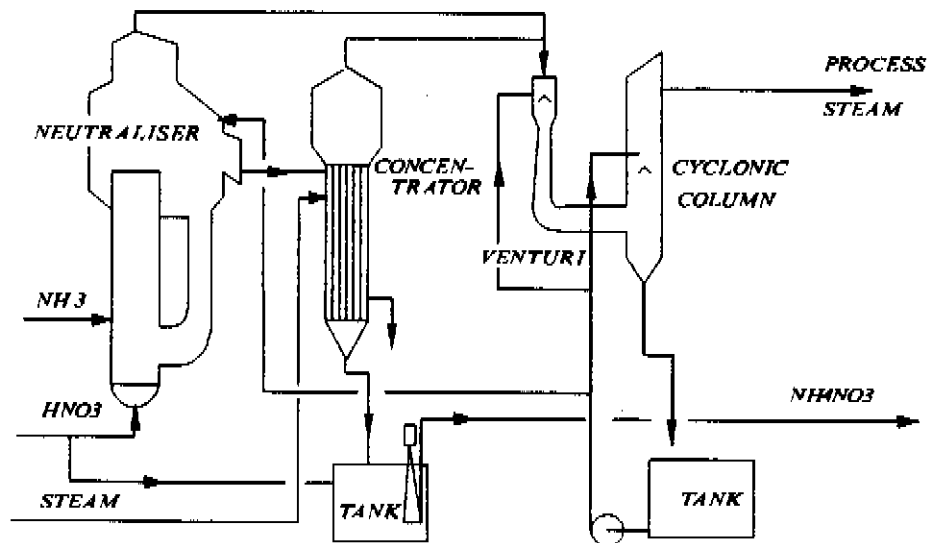
Others are more elaborated, the reaction of nitric acid and ammonia being carried out inside an intensive recycling of ammonium nitrate solution. In most cases, this recycling is performed by thermo-siphon, the reaction vessel and the recycle vessel being either concentric or separate.

The common ancestor of these recycling neutralisers, which are also used in pressure process, is without doubt the Fauser neutraliser (4), which has been developed from 1929.

The enthalpy of the steam produced in the reactor may be used to heat the nitric acid and to vaporize and superheat the ammonia. This steam may be washed in a gas scrubber, prior to utilization and discharge to the atmosphere.

Most of the non-pressure process produce a nitrate solution having a concentration, on a weight basis, of 87 % or less.

AZF has developed a process of this type (fig. 1), which is used in several of its own NPK granulation plants and in a dozen of plants of AZF's licensees (among which three plants of 700 t/day NH_4NO_3 each). This process, starting from 58 % nitric acid and liquid or gaseous ammonia, produces a 92-93 % ammonium nitrate solution, with no polluted liquid effluent, and an overall yield higher than 99 %. The investment is moderate and the plant is easy to operate and can be fully automatized.



AZF
PROCESS

Fig. 1 : AZF ATMOSPHERIC PRESSURE NEUTRALISER PROCESS

2/ The pressure process allows a better utilization of the available heat of reaction. It is their main advantage over the non-pressure process. Starting from 55 % nitric acid, it is possible, with this type of process, to get a 96 % nitrate solution. In return, the process scheme is much more complicated and the plant needs a much larger number of stainless steel equipments, resulting in a higher investment and in a less easy operation.

The pressure in the reactor is in the range of 3 to 5 bar. The temperature is most often maintained between 180 and 185° C, by the means of condensate recycling, pressure control and, in some process, by incorporating a heat exchanger producing non-polluted steam. This boiler is located either in the reactor itself or in the recycling pipe.

The reactants -nitric acid and ammonia- are most often fed into the bottom of the reaction zone. Sometimes the nitric acid is mixed with the recycled nitrate solution. Severe corrosion problems often affect the area where the nitric acid is introduced.

The process steam evolving from the pressure reactor is used to heat the nitric acid, vaporize and superheat the ammonia and to feed the vacuum heat exchanger utilized to concentrate the final ammonium nitrate solution.

A significant difficulty inherent in the pressure process lies in the production of steam and, therefore, of polluted condensates, containing ammonium nitrate and ammonia. This difficulty is increased, when the process requires to carry out the reaction in a basic pH zone, which favors the extraction of large amounts of ammonia by the process steam (the solubility of ammonia in a nitrate solution at 185° C being very low). In some process, a desorption column is provided to recover the ammonia contained in the basic condensates.

Controlling the pH of the nitrate solution is a key process point, but not a very easy one. Some prefer to measure directly the pH of the solution, as such or after dilution, but the pH-meter electrodes do not last long. Some others prefer to measure the pH of the condensates from the process steam, for the imbalance of the nitric acid and ammonia proportions immediately affects the process steam.

AZF, taking advantage of the large expertise accumulated by its ancestor companies, has developed its own pressure process (fig. 2). This process aims at reducing the well known drawbacks, already mentioned. It allows an optimal utilization of the heat of reaction, without too much complicating the general scheme and the equipments. At last, the choice of a slightly acidic pH of reaction, as well as the careful design of the layout and the equipments, limit the pollution of the process steam and the condensates to a very low level.

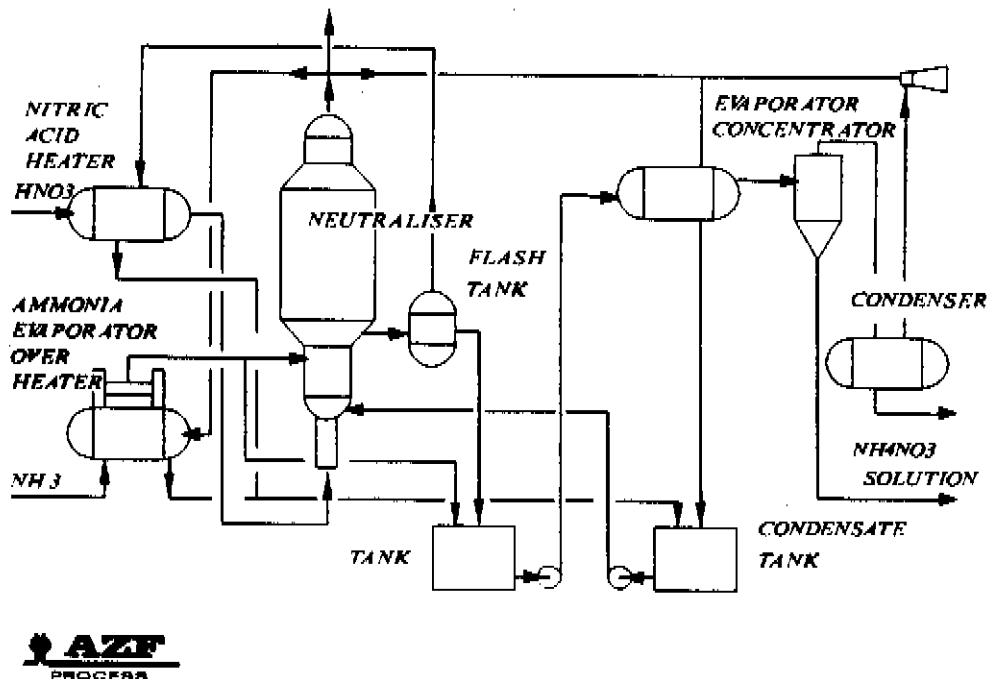


Fig.2 : AZF PRESSURE NEUTRALISER PROCESS

If we consider the simplicity of the the chemical reaction involved in the process of production of ammonium nitrate solutions, and the fact that this reaction is complete, irreversible and virtually instantaneous :



we can be surprised at the complexity and the high cost of the investments needed in practice to carry out this reaction. The evolution of the process during the past decades has even increased this discrepancy.

It is also surprising that these processes have not taken advantage of the progress accomplished in the nitric acid process, which allow, as for example the GRANDE PAROISSE process, to obtain 60 to 65 % nitric acid, with high yields and very low NO_x emissions. We even know fairly recent nitrate solution plants, using 60 % nitric acid as raw material, in which condensates are used to dilute the acid, down to 55 % !

The STENGEL process (5) which appeared in the fifties, merits a special mention, for exploring a new track : the ammonia and the nitric acid, highly preheated, react under pressure in a packed vessel of a modest size, and produce directly a 98 % nitrate solution (at 200° C) this process has had so far no industrial application.

The new AZF process, the Pipe Reactor Process, which is described below, is using a track totally different from the traditional process which we have just reminded. It allows, among other things, to take a full advantage of the possibilities available in the modern nitric acid plants.

HISTORICAL ACCOUNT OF THE NEW AZF PIPE REACTOR PROCESS

As a matter of fact, this process has originally been created to meet a need to supply our granulation pilot plant with concentrated ammonium nitrate solutions.

The operators of this pilot plant, when performing granulation tests on ammonium nitrate or NPK, had either to transport hot nitrate solutions from nearby industrial plant, or melt uncoated ammonium nitrate prills. This supply was uneasy and time-consuming.

This pilot plant was equiped with pipe reactors to make ammonium phosphates, sulphophosphates or nitrophosphates, for the well known AZF Dual Pipe Reactor Process (56 AZF pipe reactors are in operation or are being installed throughout the world) has been piloted in this plant.

The operators got then the idea to feed one pipe reactor with nitric acid and ammonia, in order to make in situ the nitrate solution that they needed. They have had, of course, to cope with the predictable difficulty of getting an important nitrate mist, but as the flows were small, that didn't constitute a real problem.

They however tried to solve it, by recycling a premade nitrate solution into the pipe reactor and by modifying some parameters (pressure, localisation of the feed mouths, diameter, length, etc...).

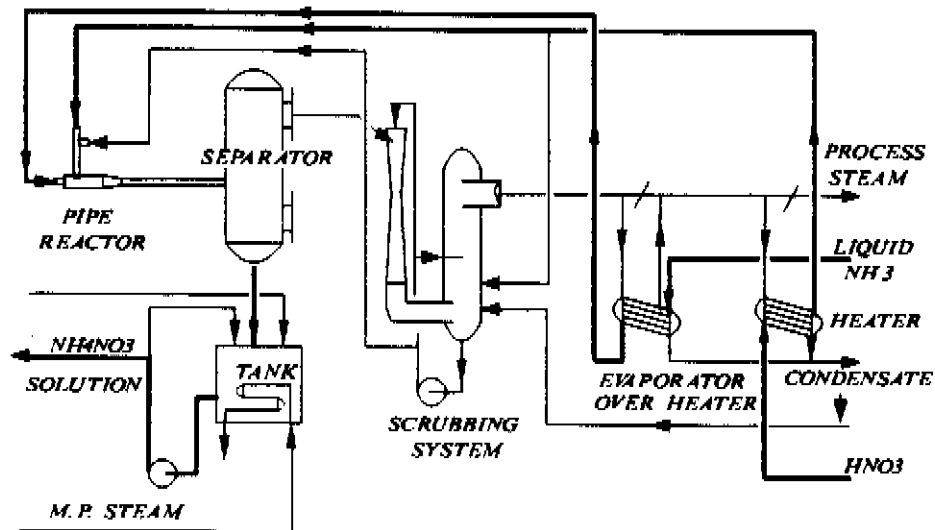
As they obtained encouraging results, the question was rapidly raised whether they could be extrapolated and the decision was then taken to develop this "convenience" into an industrial production process.

Systematic runs of tests have been carried out, resulting in the definition of the principles of this new process, which was then patented.(6)

During the fall of 1985, as we had to increase the ammonium nitrate production capacity of one of the AZF plants, located in MAZINGARBE, we took this opportunity to use the newly piloted process to carry out this extension (250 t/day). The scale-up factor of this project has been about 33, as we had to design a plant of more than 10 t/hour, starting from a pipe reactor producing 300 kg/hour.

With this first industrial application of the new process, we have been able to confirm the operation parameters defined in the pilot plant, as well as the high yield of the system. The capacity of the MAZINGARBE plant has just been increased to 500 t/day.

Soon after the start-up of the plant, one of AZF's licensees for the Dual Pipe Reactor Process, the SCOTTISH AGRICULTURAL INDUSTRIES, asked us whether we were willing to grant them a license of this new process. We have then designed a unit capable of producing 700 t/day, which started-up very easily and is fully satisfying our client.



AZF
PROCESS

Fig. 3 : AZF PIPE REACTOR PROCESS

THE NEW AZF PIPE REACTOR PROCESS (fig. 3)

Principle of the process

In the new AZF Process, the reaction $\text{HNO}_3 + \text{NH}_3$ is carried out under pressure in a pipe reactor.

The heat of reaction, in addition to its main utilization for vaporizing most of the water of the nitrate solution, is used :

- to vaporize and superheat the liquid ammonia prior to its introduction into the pipe reactor.
- to heat the nitric acid.

When starting-up , if one has imperatively to avoid a puff of mist lasting a few seconds, concentrated nitrate solution is recycled into the head of the reactor, which results in a more uniform temperature and a better mixing of the constituents.

The solution from the reactor is then flashed into an atmospheric pressure separator, in which the steam is vaporized and the droplets are separated.

This steam is then washed by a dilute nitrate solution (30 %), acidified with nitric acid, in a venturi and a cyclonic column, where the ammonia and the nitrate particles are recovered. The washed steam is finally used to heat the feedstocks.

Description of the process

1/Raw materials

Nitric acid

The nitric acid flow is regulated and, by means of a proportionometer, is linked with the ammonia flow. It is then heated to 50-60° C in an exchanger heated by process steam, prior to being fed into the pipe reactor.

Ammonia

The liquid ammonia flow, generally available at 7 bar, is regulated. The ammonia is then vaporized and superheated in an exchanger heated with process steam, prior to being fed into the pipe reactor.

2/Pipe reactor

Ammonium nitrate solution is pumped for a short while into the pipe reactor, at the beginning of the operation, only if one must absolutely avoid a puff of mist.

The reaction, initiated in the mixing zone, continues all along the reactor. The temperature obtained in the reactor is the liquid-vapor equilibrium temperature of the nitrate solution, under the reaction pressure, and at the final concentration.

3/ Separator

The nitrate solution from the reactor is depressurized into an atmospheric pressure separator.

The outlet of the separator is equipped with a demister.

4/ Ammonia vaporization

The liquid ammonia is vaporized and superheated, using process steam, in a heat exchanger. Part of the steam is condensated.

5/ Nitric acid preheating

The nitric acid is preheated in a heat exchanger, using process steam. In case 63 % nitric acid is available and if one does not need a nitrate solution richer than 97 %, this operation can be omitted.

6/ Nitrate solution buffer tank

After flash depressurization into the separator, the nitrate solution flows by gravity into a buffer tank, where it is pumped to be utilized. This pump is also used to recycle solution into the tank, in order to homogenize the nitrate.

A small flow of gaseous ammonia is introduced into the buffer tank, to complete the neutralization. A pH-meter automatically controls this flow.

7/ Steam scrubbing

Remark : this operation is necessary only if a 99,9 % yield is required, or if the local environment regulations are particularly severe.

The basic steam from the separator, which may contain minor quantities of ammonia and nitrate droplets, passes through a venturi, fed with an acidified 30 % ammonium nitrate solution. The pH of this solution is controlled.

The ammonia recovery is completed in a cyclonic column, fed with the same solution.

A demister perfects the cleaning of the steam.

The washing solution from the column is pumped into the pipe reactor. This flow is regulated. An introduction of condensates into the bottom of the column maintains a constant level.

This addition of water allows to :

- desuperheat the basic steam
- absorb the neutralization heat of the ammonia
- adjust the nitrate concentration of the washing solution

The clean steam from the column passes through the heat exchangers and, at last, is either discharged to the atmosphere, or condensed, or used in downstream plants, such as granulation or "prill fattening" plant.

8/ Condensates recovery

The clean condensates from the various heat exchangers go to a buffer tank, and are used to feed the washing column and in the nitric acid plant. The surplus is discharged into the sewage system.

9/ Block diagram (fig. 4)

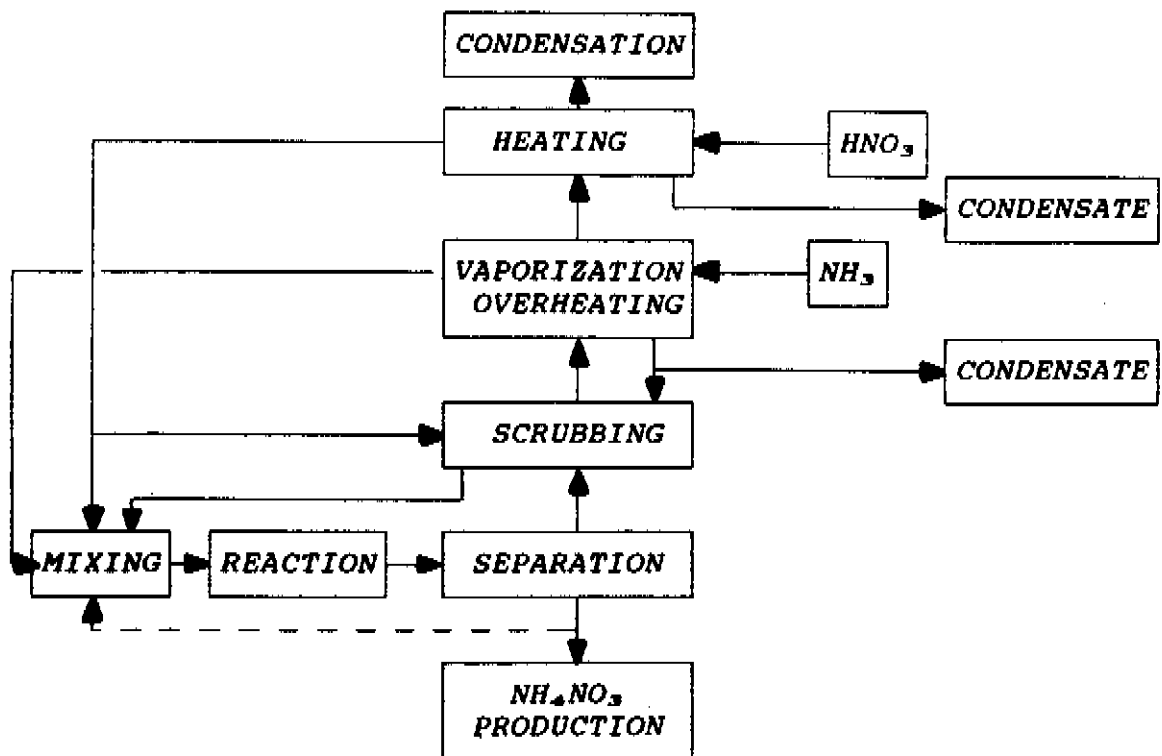
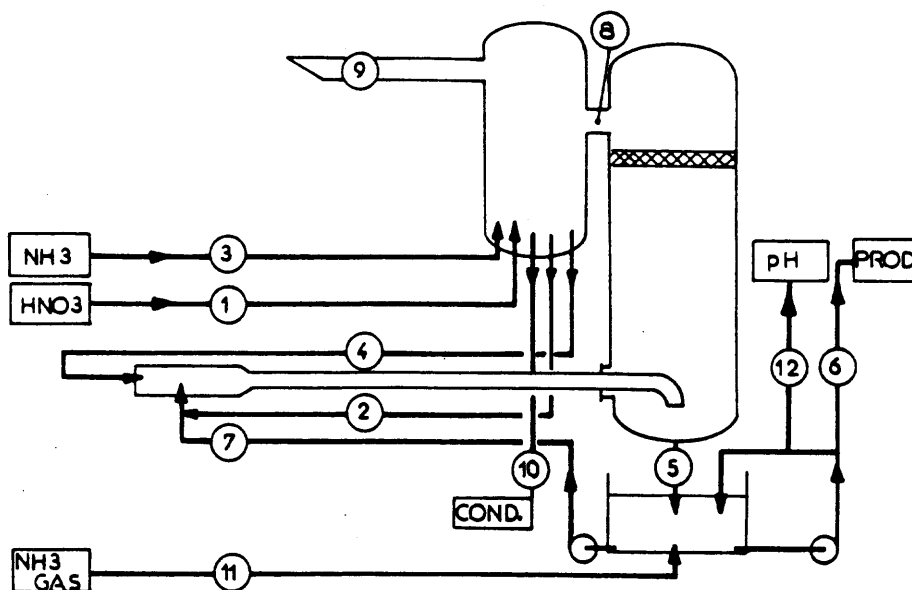


Fig. 4 : BLOCK DIAGRAM

10/ Operating data

The table 1 shows in detail the operating data of AZF first industrial unit, in MAZINGARBE. This set of data was measured during the program of tests of April 1988. The actual production of solution was 21 t/h, expressed as 100 % NH_4NO_3 . In this unit there is no steam washing section.



kg/h	NH_3	Na	HNO_3 100 %	Na	$\% \text{HNO}_3$	NH_4NO_3 100%	$\% \text{NH}_4\text{NO}_3$	H_2O	Temp.	Volume flow- rate	pH	free HNO_3 g/l
1	-	-	16823.5	3758.56	57.71	-	-	12328.3	37.00	22.00	-	-
2	-	-	16823.5	3738.56	57.71	-	-	12328.3	57.20	-	-	-
3	4516.44	3719.42	-	-	-	-	-	-	5.20	6.85	-	-
4	4516.44	3719.42	-	-	-	-	-	-	79.50	-	-	-
5	0.00	3686.54	42.10	3695.90	-	21065.94	91.04	2030.11	154.00	16.84	2.45	2.50
6	3.37	3698.68	0.00	3695.90	-	21119.48	91.21	2030.11	149.70	16.78	4.70	0.00
7	0.00	0.00	0.00	0.00	-	0.00	91.21	0.00	149.70	0.00	4.70	0.00
8	0.00	32.88	44.00	42.66	-	187.90	-	10298.2	156.00	-	-	-
9	0.00	28.78	18.17	24.82	-	118.76	-	7583.17	183.00	-	-	-
10	0.00	12.18	25.79	17.83	-	69.14	2.46	2715.83	98.00	2.76	1.4	9.34
11	14.74	12.14	-	-	-	-	-	-	27.00	2.24	-	-
12	0.00	0.00	0.00	0.00	-	0.00	91.21	0.00	149.70	-	4.70	0.00

MAZINGARBE UNIT - TEST RUN
APRIL 1988 - 21 t/h

Table 1 : OPERATING DATA FOR EACH PROCESS LINE

The table 2 gives an average balance of this unit.

<i>MAZINGARBE UNIT - TEST RUN - APRIL 1988 AVERAGE BALANCE</i>		
<i>Free acidity reactor outlet (Free HNO₃) g/l</i>		<i>2.50</i>
<i>Recycle ratio</i>	$\frac{\text{Vol. recycled nitrate}}{\text{Vol. HNO}_3} \times 100$	<i>0.0</i>
<i>Total Nitrogen losses</i>	$\frac{\text{Total Nitrogen lost}}{\text{Total Nitrogen engaged}} \times 100$	<i>1.01</i>
<i>Ammoniacal N losses</i>	$\frac{\text{Ammoniacal N lost}}{\text{Ammoniacal N engaged}} \times 100$	<i>0.88</i>
<i>Nitric Nitrogen losses</i>	$\frac{\text{Nitric N lost}}{\text{Nitric N engaged}} \times 100$	<i>1.14</i>
<i>Maximum temperature °C</i>		<i>186.0</i>
<i>Absolute pressure at reactor head atm.</i>		<i>5.6</i>

Table 2 : AVERAGE BALANCE

CONCLUSION

The new AZF process of production of ammonium nitrate solution by a pipe reactor successfully opens a new track in this important industrial field.

The process has significant advantages over the traditional process, in many aspects.

1/ Safety

The risks of explosion of a pure ammonium nitrate solution, non contaminated by reducing agents or by decomposition catalysts, are practically nil, except if a confined solution is heated at very high temperatures, about 300°C (7).

However, a decomposition hazard appears when a large volume of acid solution is kept for a rather long time, the acidity of the solution increasing spontaneously (8).

The new AZF process completely eliminates this risk, since the reaction mixture volume is extremely low -a few tens of liters for a production capacity of 1 000 t/day- and since the residence time in the pipe reactor is in the order of magnitude of a second.

2/ Easy operation

As the new AZF process requires a small number of equipments, and as the reactor volume is very small, a stable operation is very rapidly reached.

The start-up and the shut-down of the plant, which can be easily automatized, are quite instantaneous.

3/ High flexibility

The design of the AZF pipe reactor, as well as the other equipment, allow a high flexibility (the rate of production can be varied in the range of 1 to 3), which is not the case with the conventional pressure process. We think that this flexibility can still be increased and we are presently working on it.

4/ Direct production of highly concentrated solutions

From 58 % nitric acid, it is possible, without any concentrator, to obtain directly a 92 % solution.

When 63 % nitric acid is available, which is now usual in the modern plants, it is possible to reach a concentration of 97 % or even more. Studies are in progress to reach 98.5 - 99 %.

5/ Low investment and maintenance costs

A comparative study that we have performed has shown that the investment cost of a 1 000 t/day unit represents, with the new AZF process, half of the cost of a conventional pressure plant.

This is one of the reasons, and not the least, of choosing this new process for the 1 000 t/day plant, which is presently being built at the ROUEN site of GRANDE PAROISSE.

The pipe reactor is the only piece of equipment on which we expected maintenance costs. We had planned to replace periodically some parts. As a matter of fact, since the MAZINGARBE plant started, at the end of 1985, we have had to do no maintenance work on this plant. We have just replaced the pipe reactor to increase the capacity of this plant from 250 t/day up to 500 t/day.

References :

- 1/ G. D. HONTI - "The Nitrogen Industry"
Akadémiai Kiado - BUDAPEST
- 2/ V. SAUCHELLI - "Fertilizer Nitrogen"
ACS Monograph n°161
Reinhold Publishing Corporation
- 3/ C. KELETI - "Nitric Acid and Fertilizer Nitrates"
Fertilizer science and technology series
volume 4
- 4/ FAUSER - RDP 590 469 (1931)
- 5/ HESTER, DORSEY, KAUFMAN - "STENGEL Process Ammonium
Nitrate"
Ind. Eng. Chem (1954)
- 6/ European Patent - 86903437-1 (1986)
- 7/ K.D. SHAH and A.G. ROBERTS, ICI, - "Safety Considerations
in the processing, handling, and storage
of ammonium nitrate"
C. KELETI "NITRIC ACID AND FERTILIZER
NITRATES" p. 261-273.
- 8/ C.H. SOLOMON, K.S. BARCLAY - "Ammonium Nitrate :
Manufacture and use"
The Fertiliser Society - Proceedings n° 85

TA/88/9 A new process for the production of concentrated ammonium nitrate solutions by P. Chinal, G. Cousserans, J.B. Peudpiece and J.F. Priat, Société Chimique de la Grande Paroisse, France

DISCUSSION (Rapporteur Mr. A.F. Porneuf, Société Chimique de la Grande Paroisse, France)

Q - Mr. A. HAMDJ (ICM/SAEPA, Tunisia).

1/ Can you adapt a pipe reactor to an existing plant without too many alterations in order to lower the technical minimum and increase production ?

2/ With a 58% nitric acid solution, can you obtain a 95-96% ammonium nitrate solution ?

3/ What is the order of magnitude of investments for a 500 t/d unit ?

4/ What is the reason for the investment being 50% lower than for a conventional unit ?

A - 1/ The pipe reactor can indeed be adapted to an existing plant and we have projects under investigation to implement that possibility. The existing equipment on which the pipe reactor will be grafted must have a sufficient size, but this is normally the case. One can generally use the neutralizer of an old plant as a separator by grafting a pipe reactor on it.

2/ Can we obtain a 95-96% solution with 58% nitric acid ? No, I think that today we can obtain a 93-94% solution according to the temperature of the acid. It is the limit because, if we try to heat up the nitric acid too much, corrosion problems appear. Then with the technology and the materials available today, we do not think we can go higher. But, in a second stage, the one we are now investigating, we may succeed in gaining 1 or 2% concentration without heating the nitric acid any further.

3/ I do not remember the figures for the investment in a 500 t/d plant. However, the cost for a 1000 t/d plant amounts to about US \$ 2 million. But it is always risky to announce such figures since local conditions are always an important factor.

4/ Why is the investment cost 50% lower ? It is mainly due to the weight of stainless steel used. A pipe reactor weighs a few tens of kg. A conventional saturator or a neutralizer can weigh 2 tons or more. In addition, circuits are much more complicated in a conventional plant. There are many more exchangers, then much more stainless steel and engineering. This is not a figure we have estimated. When our management decided the construction of a new 1000 t/d plant at our site in Rouen, we compared the two processes; the conventional process was almost double.

Q - Mr. B.K. JAIN (FAI, India).

Considering the safety aspects, what will the capacity restriction be of the new AZF pipe reactor ? Kindly indicate the construction materials used for the pipe reactor and the expected life under normal conditions.

.../.../...

A - The material used for the whole plant, except for the nitric acid heater - if it exists, which is not always the case - is 304 L type stainless steel. The lifetime was mentioned to you in relation to our industrial experience at Mazingarbe. We had a pipe reactor which was two years in operation and was then replaced not because it was corroded, but because we wanted to produce more. I cannot tell you that it lasted for 10 years, because we do not have 10 years of experience, but two years for sure. In addition, I do not think that replacing a pipe reactor is a serious problem. It is only a piece of pipe.

Q - Mr. J.D. CRERAR (Norsk Hydro Fertilizers, U.K.).

It surprised me a little to see the title of this paper: "The new AZF process for ammonium nitrate", because it seems to me that the process has many similarities to the Stengel process to which you refer and to the improved Stengel process which we have operated in England for about thirty years. The only difference, as far as I can see, is the fact that your reactor is horizontal compared with a packed vertical reactor.

1/ The recoveries of nitrate and ammoniacal nitrogen, which you have quoted, are unlikely to be acceptable to most industrialized countries and, therefore, the optional scrubber which you mentioned is very likely to be required. Could you give me the ammoniacal and nitrate content of the condensate from your process when that optional scrubber is included, please ?

2/ Could you tell me which of those plants that you listed in France, in Scotland and in Australia, have the scrubber fitted ?

A - Concerning your remarks about the Stengel process, I think there is more difference than you mention, since, in that process, there is an internal packing inside the reactor. In the original Stengel process, as I know it, there was a hot air steam in the separator to reach the expected concentration. But I do not believe it is worthwhile starting a debate on the Stengel process.

Concerning the ammoniacal and nitric nitrogen in the condensate, I think you have the answer in the written paper. Indeed, there is no steam scrubbing in the plants at Mazingarbe and in Scotland, since it was not necessary in order to reach the 99% concentration requested.

However, we are installing one in our plant under construction at Rouen. It is actually a steam scrubbing system we have transposed from our old atmospheric pressure process and a few are operating in our plants. There are five working in the USSR, but I do not have the reference list. It is not only something on paper, but something which actually operates and which we simply transposed from our old process because we were satisfied with it.

Q - Mr. G. JUIF (Technip, France)

What is the maximum capacity we can expect from one single pipe reactor ?

A - At present, I would say 1000 t/d. I feel we should be able to reach 1500 t/d with one single pipe reactor. If we had to build in one of our plants a 1500 t/d unit, I think that today we would take the risk

.../.../...

of building it with our new process.

Q - Mr. L. RASMUSSEN (Superfos Fertilizers, Denmark).

1/ Could you please tell us the yield you have been using in the cost comparison mentioned on page 12 in your paper ?

2/ If a yield of 99 or 99.5% is required, would that affect your comparison dramatically ?

3/ What is the turndown ratio of the process ?

A - 1/ In the comparison of investment costs mentioned on page 11 of our paper, we assumed that the efficiency of each plant was the same. We compared our pipe reactor process including gas scrubbing with another process under pressure also including gas scrubbing. The conditions are the same.

2/ I do not know how to answer your second question, because it is possible to reach 99-99.5% efficiency theoretically. However, I think that, to measure the efficiency, we must measure the losses very carefully.

Q - Mr. G. KONGSHAUG (Norsk Hydro, Norway).

Can you indicate the retention time and flow speed in the pipe reactor ?

A - The retention time in the pipe reactor is less than one second. The speed depends on the flow coefficient per square cm adopted. It differs if we operate in the lower part of the possibilities of the pipe reactor or, conversely, close to the maximum, and this depends on the operational pressure. But the speed inside the pipe reactor is about 200 m/sec. It is difficult to answer that question because feedstocks, such as nitric acid, which enter in liquid form, have a low speed. Ammonia enters in the gaseous form at a relatively higher speed. Then the reaction takes place. Part of the water in the nitric acid evaporates gradually when progressing in the pipe reactor and the speed increases. Then it depends at what point the speed is actually measured and, at present, we are not able to measure it. It can only be evaluated.

Q - Mr. P. ORPHANIDES (Duetag, France).

1/ The first question concerned the material of the pipe reactor, but it was already answered. The nozzle is also in ordinary 304 L ?

A - In fact, we did two experiments. On one pipe reactor we used a higher quality and more expensive material. But, in view of the results obtained, the 304 L is the adequate material.

2/ What are the improvements to be expected in revamping an existing plant with atmospheric neutralization applying your process in respect of higher efficiency and increased concentration capacity ?

A - I cannot give you a general answer, since it depends on the type of process we have to operate with. I have in mind conventional processes working very well, but where the potential of concentration of nitrate produced is somewhat wasted, because, for reasons difficult to understand, the solution is often rediluted during production. I know

an old plant where 60% nitric acid was produced and where, in fact, we realized that this acid was used at 55%, because it was diluted with condensates. In other plants, it is not the same thing and, in that case, I think we can gain concentration capacity e.g. that normally with 58% acid 92-93%, even 94%, nitrate can be produced according to the temperature at which the nitric acid is heated.

3/ What is the concentration to be expected in the case of 60% acid, when, as indicated in your flowsheet, liquid effluents from the scrubber are introduced in the reactor ?

A - When we introduce again the steam scrubbing solution in the pipe reactor, we lose about 1% concentration.

Q - It means that with a preheated acid of 60% maximum concentration after recycling, what nitrate concentration do you reach ?

A - I do not have the balance, but about 95%.

Q - If you continue recycling ammonium nitrate produced, as you do during the starting stage, can you improve the final concentration ?

A - No. In fact we do not recycle anymore, since the plant starts immediately after we press the button.

Q - What is the concentration of the scrubbing solution ? Is there no risk of crystallization ?

A - The ammonium nitrate solution used as scrubbing solution contains 30% nitrate and there is no risk of crystallization. When the plant stops, it is not emptied. It needs no dilution when restarting.

Q - My question concerned the recycling of part of the concentrated solution, 95%, to gain a little ?

A - It is the way we started operating the process, because we did not dare, in the first place, to have nitric acid and ammonia alone in the reactor and we began to recycle a previously produced nitrate solution. As a result, there were three inlets in the pipe reactor for nitric acid, ammonia and recycled nitrate solution. We realized that with a recycling of less than five we managed to avoid the mist. But that recycling did not satisfy us and we tried to gradually reduce the recycling by changing the operating conditions in the pipe reactor and we realized we could completely do without recycling.

Q - Mr. H. HERO (Kemira Oy, Finland).

1/ Your process shows very high yields and high capacities, but can you give us some figures of your nitrous values after a pipe reactor, and in your stack gases ?

2/ What has been your experience in using phosphoric acid together with nitric acid in your pipe reactor ?

A - 1/ I shall answer your first question in writing because I do not have the data here.

.../.../...

2/ Concerning the use of phosphoric acid in our pipe reactor, we have a long and old experience since, at present, there are 56 pipe reactors operating or under construction in the world following our process. It is indeed because we have the experience of making ammonium phosphate or sulphate-phosphate in a pipe reactor that people in our company had the idea, because they needed ammonium nitrate solutions, to use a pipe reactor available in our pilot plant. We also used a mixture of phosphoric acid and nitric acid in a pipe reactor in a granulation plant.

Q - Mr. V. BIZZOTTO (NSM/Norsk Hydro, Belgium).

Page 9 of your presentation you stated that ammonium nitrate solution process surprisingly did not take advantage of the modern nitric acid plant capacity of producing 60-65% acid. May I just inform and remind you that NSM did develop its own ammonium nitrate solution process using the maximum available concentration of 62-63%.

The first industrial plant, 2,000 t/d, went into operation in 1977 at Sluiskil in the Netherlands. The second one, 3,300 t/d in 1985 at Sluiskil and the third one, 3,000 t/d in 1987 at Immingham. All three were under pressure with a clean-steam production, zero pollution, 96-97% final concentration, no corrosion.