

MAP MANUFACTURE USING THE PIPE REACTOR

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ABSTRACT

Texasgulf, at its Lee Creek, N.C. facility, has converted its Dorrco GTSP plant to alternate production of MAP, 11-52-0, or 11-55-0. The use of a TVA 10 inch Pipe Reactor has resulted in high ammoniation efficiencies, low emissions of fluorine and ammonia and completely satisfactory products. The Pipe Reactor, currently not used widely for MAP at capacities above 50 TPH, has advantages in being able to use high strength acid, resulting in low recycle and high capacity, with emissions well below Florida and EPA Standards.

At Texasgulf, relatively low strength acid (38% to 43% P₂O₅) is used. In this case, virtually all the available reaction heat is released into the granulator to raise the granulator temperature and maximize moisture elimination.

The paper discusses the current state-of-the-art in Pipe Reactor processing, particularly in North America, compares alternate processes for MAP and discusses the operation of DAP using the Pipe.

INTRODUCTION

The use of a Pipe Reactor to make MAP grades, such as 11-52-0, is not widely practiced in the U.S.A. However, there are some significant advantages for using the Pipe to make MAP, particularly. We would like to discuss these in this paper, and compare the operation using the Pipe with that of other MAP processes.

In 1986, Texasgulf began producing, on a campaign basis, 11-52-0 MAP using a 10" TVA Pipe Reactor in their Dorrco GTSP plant. The Pipe used by Texasgulf is very similar in size and design to one used by Royster to make over 300,000 tons of 18-46-0 DAP during 1986 and 1987.

The successful MAP operation at Texasgulf is based on the excellent ammoniating efficiency achieved in the pipe. This reduces the amount of acid required in the scrubber system to little or none, and reduces the fluorine evolved in the process, so that emissions of both ammonia and fluorine are low.

The Pipe Reactor, operating at temperatures of 280°F to 330°F, and pressures of 30 to 50 psig, provides a means of making MAP, and MAP-sulfate grades (e.g. 16-20-0) at lower moistures, lower recycle and higher capacities.

PIPE REACTOR PROCESSING

MAP Pipe Reactor Process Modes

A typical flowsheet, Figure 1, shows the addition of a Pipe Reactor to a slurry process GTSP plant. The retrofit of a Pipe Reactor to such a facility at Texasgulf's Aurora, N.C. plant will be described in more detail later in this paper.

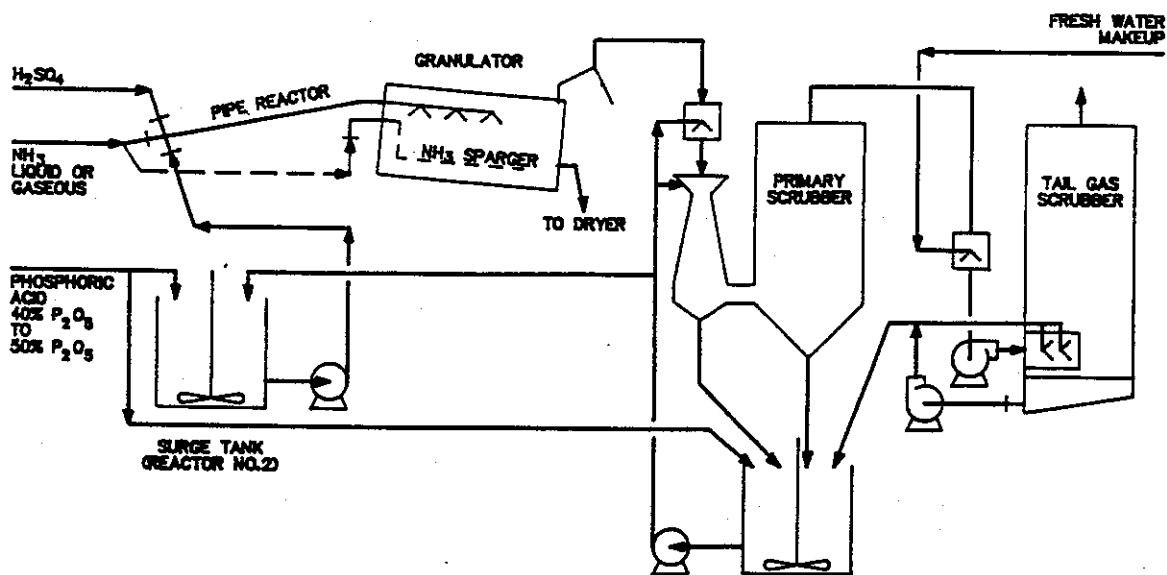


Fig. 1 PARTIAL FLOWSHEET PIPE REACTOR MAP PROCESS

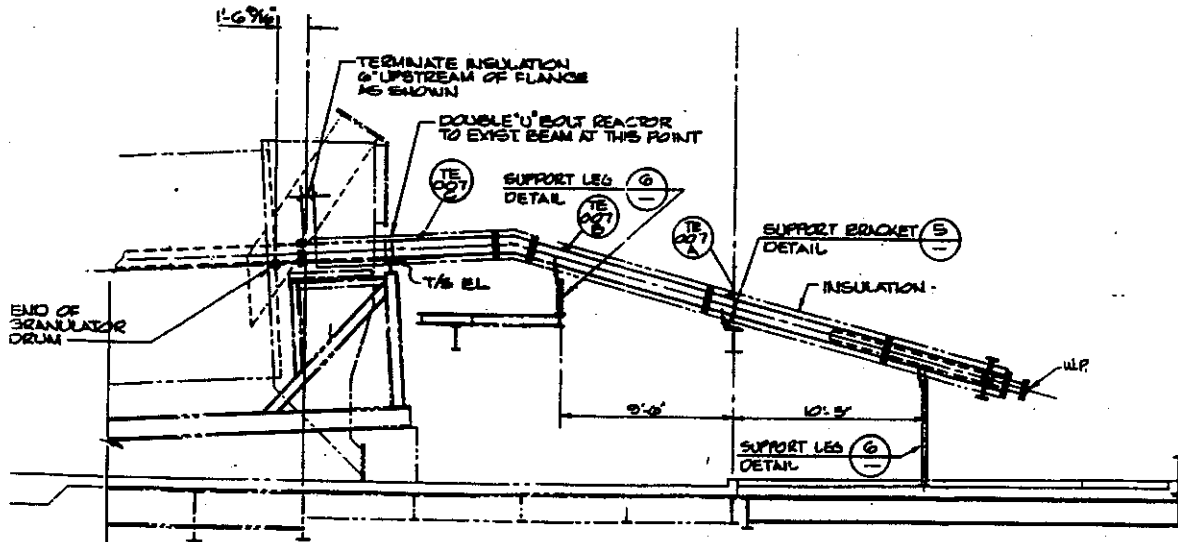
At least two options exist in processing MAP with the Pipe. The simplest procedure is to do all the ammoniation in the Pipe itself. This results in very little ammonia slip to the scrubbing system and ultimately to very low emission of both fluorine and ammonia.

The second option employs the conventional ammonia sparger shown dotted in Figure 1, so that some ammoniation is done in the granulator. In general, the extent of sparger ammoniation is limited to avoid excessive NH_3 slip. In this mode, ammoniation is carried out to 0.7 to 0.8 mole ratio in the Pipe Reactor and completed using the ammonia sparger. More phosphoric acid is required in the scrubbing system and therefore, fluorine and ammonia emissions are likely to be somewhat greater. However, the potential for higher capacity exists in the second option, because the ammoniation is being spread over both the Pipe and the sparger. This method also may result in significant citrate insoluble P_2O_5 in the product with some feed acids.

Pipe Reactor Design

The Pipe Reactors installed at Royster and at Texasgulf incorporate TVA design details including an inclined reactor pipe of suitable length and a distributor extension. A typical general arrangement of a 200mm (10") \emptyset Pipe Reactor is shown in Figure 2.

The Pipe is long enough to accomplish complete reaction. This can be monitored by the temperature rise as sensed by the three temperature elements shown in Figure 2. Pipe reactors can be provided from 100mm (4") to 300mm (12") in



diameter, spanning the range of 20 STPH to 120 MTPH MAP, accommodating acid strengths from under 40% P_2O_5 to 54% P_2O_5 , or higher.

Hastelloy G-30 is the preferred material of construction for high sulfate grades like 16-20-0 or NPK's where chlorides from the scrubber system could be present. For 11-52-0, 10-50-0 or 18-46-0 a combination of sections made of Ferralium, Jessop 700 (UB-6) and 317 is recommended. The use of Teflon or similar linings can also be satisfactory.

Conversion of Slurry TSP Plants to MAP Production

There is an increasing market for MAP 11-52-0, as a base for bulk blending, especially where granular urea is available, and as a base for suspension fertilizers. As a 10-50-0 grade, it provides an outlet for sludge produced in MGA (Merchant Grade Acid) Clarification.

Many slurry TSP plants were built without MAP or DAP capability. The addition of a Pipe Reactor to such facilities provides increased product flexibility, often at moderate add-on cost.

In retrofitting MAP/DAP Pipe Reactor operation to a TSP plant, the No. 2 TSP reactor serves as a surge tank to collect scrubber solution and phosphoric acid feed. The use of H_2SO_4 is optional. It can be provided to adjust product grade, or to increase pipe reactor temperature in the processing of MAPP (Mono ammonium polyphosphate) containing up to 15% of the P_2O_5 in the non-ortho form.

The scrubbing system shown in Figure 1 is one option. The system can also be run on once-through or recycled pond water in the tailgas scrubber.

When the ammoniation is done solely in the pipe reactor, little or no phosphoric acid is required in the scrubbing system. The MAP dust, which has a pH of about 4.5 to 5.0, dissolves in recycled fresh water to absorb a substantial portion of the ammonia slip from the Pipe Reactor. Fresh water advance is about 5 gpm to 25 gpm depending on the plant throughput and cyclone efficiency. The quantity of water required is equivalent to a reduction of about 1.5% in composite acid P_2O_5 strength.

CONVERSION OF THE DORRCO GTSP LINE AT TEXASGULF'S LEE CREEK PLANT

TSP Plant Description

Texasgulf operates a GTSP plant utilizing the Dorrco Slurry process at Lee Creek. The plant is pictured in Figure 3. In 1985, a project was begun to modify the plant to produce various grades of MAP on a campaign basis. The conversion was completed in 1986, and MAP, 11-52-0 primarily, has been produced since that time on a campaign basis between TSP operation.

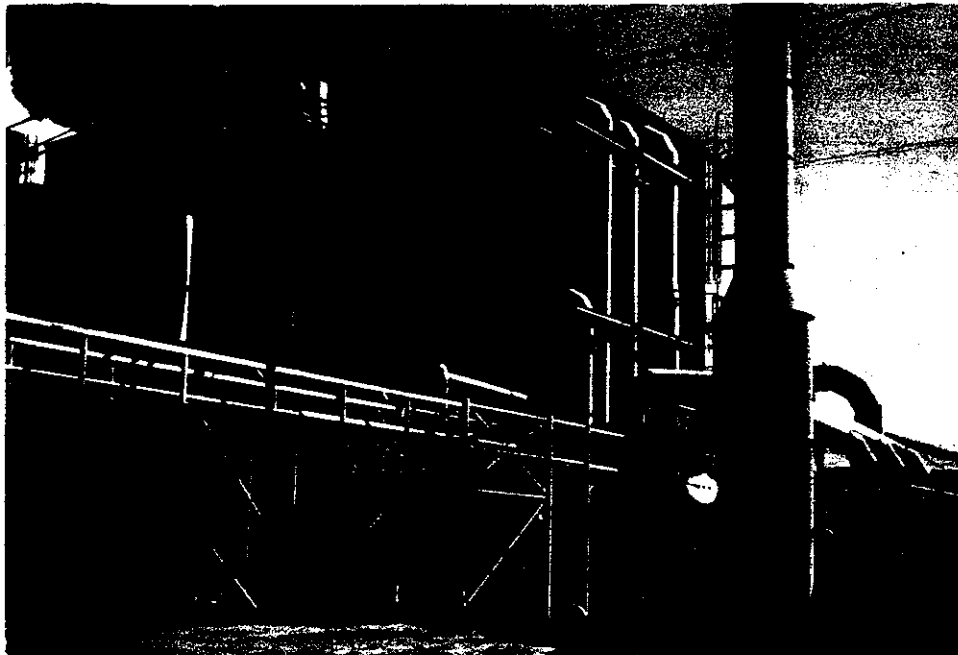


Fig. 3 TEXASGULF GTSP PLANT, LEE CREEK, N.C.

The plant, which started TSP operation in 1974, has two rock digestion reactors in series, with no preneutralizer. The scrubbing system consisted of sprayed cyclonic scrubbers operating on once through pond water, followed by a tailgas scrubber operating on recycled fresh water.

The plant has a conventional rubber lined drum granulator. TSP reaction slurry is fed by centrifugal pumps to a slurry header in the granulator.

Major Elements of the Conversion

The major elements of the conversion were:

- (1) Modification of the scrubber system to provide (a) a venturi ejector-type scrubber for the granulator fumes, (b) a scrubber sump tank with collection pipes from the several primary scrubbers, together with scrubber solution recirculation pumps and (c) a new, larger, spray nozzle system to replace multiple small nozzles which were prone to plugging and difficult to pull and clean. The modified scrubbers system is shown in Figure 4.
- (2) The addition of ammonia piping and metering, including an ammonia vaporizer, to help compensate for the relatively low strength (38% to 43% P_2O_5) phosphoric acid available.
- (3) Larger granulator slurry pumps consistent with the higher rate of operation on MAP.
- (4) The Pipe Reactor itself including steam supply and all feed connections.

Performance of the Pipe Reactor at Texasgulf

The plant has produced MAP, primarily 11-52-0, during campaigns of up to 3 weeks in duration, at rates up to 70 STPH. The normal rates for GTSP are 45 to 50 TPH.

The Pipe Reactor itself appears to have a capacity substantially in excess of 70 TPH MAP because the temperature probes read a fairly constant value following the mixing Tee. At phosphoric acid concentrations above 46% P_2O_5 , the plant is projected to have well over 90 TPH capacity. However, no modifications have been made, to date, to the product cooling system, originally sized for 50 TPH, so that capacity is limited to about 70 TPH at present.

The Pipe Reactor, which is made from Hastelloy G-3 (predecessor of G-30) has shown no wear and experienced little scaling. The pipe is steamed out whenever production stops and during scheduled down days. No other special cleaning has been required.

The pipe operates at 30 psig to about 50 psig depending on rate, and at about 320°F to 330°F. Recycle is about 4.5:1 to 5.0:1, when using 38% to 43% P_2O_5 acid. The plant consumes fuel at these phosphoric acid concentrations at a rate of about 250,000 to 400,000 BTU/short ton of product.

Product quality was the subject of a paper⁽¹⁾ by John Jernigan of Texasgulf. This paper covered the effect of different fillers on the product. Since the Texasgulf acid is relatively pure it can produce an 11-55-0 grade, therefore filler or sludge is added when the 11-52-0 grade is produced.

Texasgulf's product specification for 11-52-0 is 95% in the minus 6 plus 14 (Tyler) mesh range. The SGN (size guide number) is 220 to 230.

The product has a low dust content and a moisture below 1.0%.

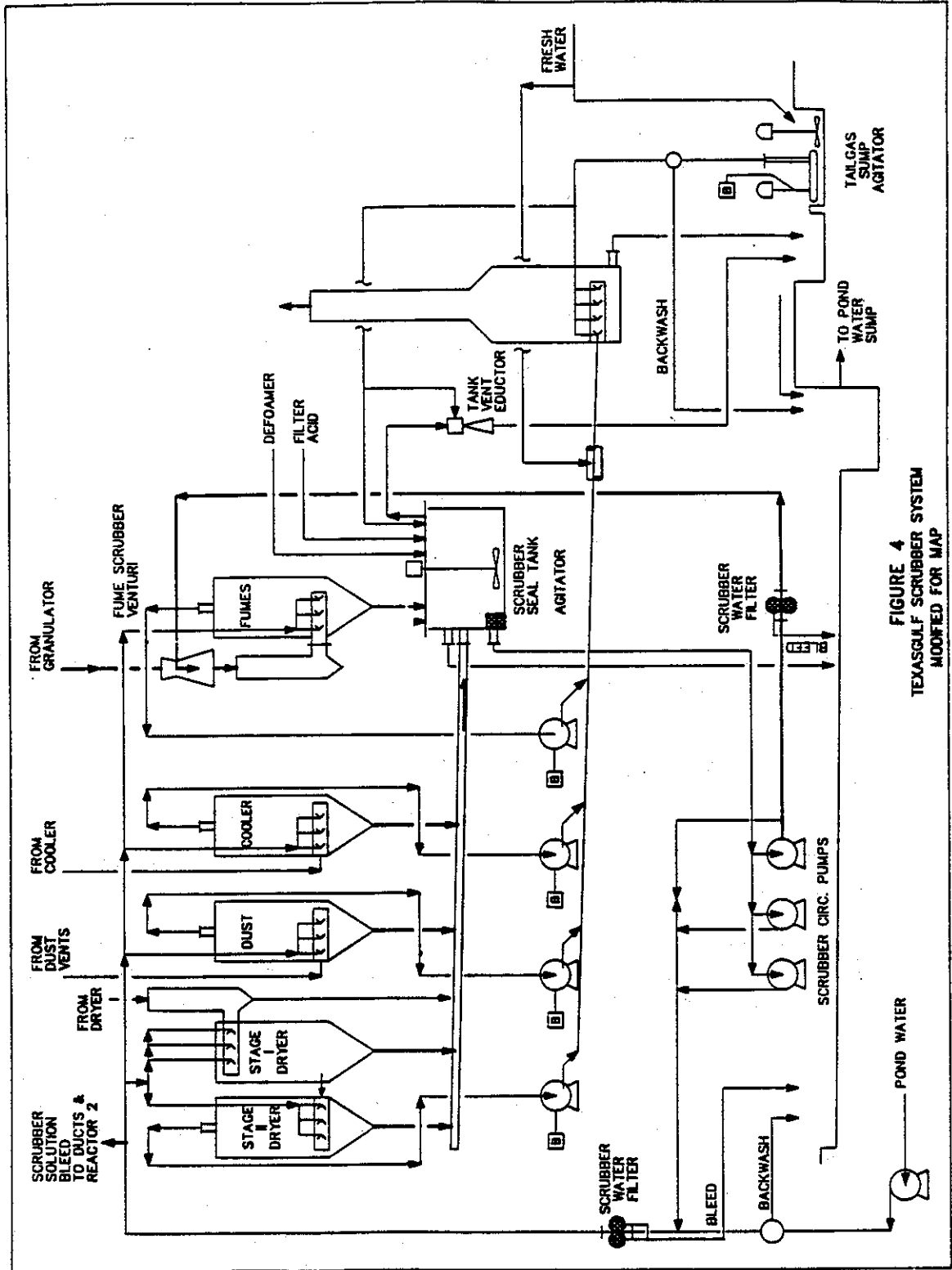


Fig. 4 TEXASGULF SCRUBBER SYSTEM
MODIFIED FOR MAP

In the initial MAP operation, the scrubber solution tended to dissolve TSP scale which had accumulated over 10 years of operation and, in spite of line strainers, severe scrubber nozzle plugging occurred. This was alleviated by the nozzle modification previously described and has worked well.

After a campaign on MAP, a very thorough cleaning, even washing, of the plant is required. A small quantity of ammonia in TSP results in a very sticky mess. Small quantities of TSP in MAP present no such problem.

Texasgulf has two other lines which produce DAP so no trials have been made thus far to produce 18-46-0 in the Pipe Reactor line. However, the first stage scrubber on the granulator fumes is now a venturi-ejector with a very high liquid-to-gas ratio and this, together with the spray nozzle modification described above, would suggest that DAP can be produced efficiently. Another factor in favor of successful production of 18-46-0 using the Pipe Reactor in this plant, is the relatively high purity of the acid available from North Carolina rock. This makes it possible to make an 18-46-0 grade at a slightly lower mole ratio than when using Florida acid.

The plant is designed to produce 10-50-0, 11-52-0, 13-52-0, 12-55-0 (ammonium poly-phosphate) and 11-55-0.

Texasgulf has also operated smaller TVA Pipe Reactor installations at its Kinston, N.C. granulation plant and at Lee Creek in their No. 1 DAP facility. At Kinston, 13-54-0 and 12-55-0 have been produced, based on imported merchant grade phosphoric acid. These grades, and even 11-55-0, have somewhat higher total plant food content than conventional DAP, and can be produced economically from Texasgulf Merchant Grade Acid, using a Pipe Reactor in a conventional granulation facility, for bulk blending or direct application products.

Performance of the Pipe Reactor Making MAP from Florida Acid

The operation of the similar 10" Pipe Reactor at Royster on MAP is described by Salladay and Paulson⁽²⁾. In this case, operating rates of about 60 TPH were run making products of 11.0-50.0-0 to 11-53-0, using a composite acid strength of 47% P₂O₅. The operation required no fuel.

In processing Florida acid in Pipe Reactors, the perceived risk is that scaling will occur due to sludge containing iron and aluminum solids. However, in the Royster MAP Pipe Reactor operation, the objective was to dispose of the sludge previously fed to a TSP operation which management sought to eliminate. The acid contained as much as 17% to 18% solids. Some scaling did occur in the Pipe at the point where there is a change of slope, but more scale formed as a growth at the discharge slot. In the Royster case, the scaling was controlled by steaming and periodic washing with pond water. At Royster, no sulfuric acid was fed to the Pipe; TVA's experience is that H₂SO₄ to the Pipe reduces scaling.

ADVANTAGES OF THE PIPE REACTOR FOR MAP PROCESSING

Excellent Ammoniation Efficiency and Low Fluorine Emission

The Pipe Reactor has excellent ammoniation efficiency showing low ammonia slip when operating in the 1.05 to 1.1 mole ratio range; 2% to 3% NH₃ slip is a normal

figure. This results in the need for very little or no phosphoric acid in the scrubber system. The Pipe Reactor itself evolves relatively low quantities of F, estimated at about 300 to 400 lbs. per day at 65 TPH. This is relatively easily collected. Fluorine can be a problem in other MAP processes as discussed later.

The Pipe Reactor Can Use Highly Concentrated Acid

Because the Pipe Reactor operates at high temperature, 280°F to 330°F in MAP processing, the slurry remains fluid at low moisture levels, and the low solubility of MAP is overcome. This reduces the moisture level in the system, results in higher granulator temperatures, low recycle requirements and high operating capacities. Using acid in the 46% to 50% P₂O₅ range, the operation is autogenous, requiring no fuel for drying. The Pipe Reactor has the advantage of utilizing much higher composite acid strength than the "Back Titration" process and higher even than the "Granulator Ammoniation" method. These MAP processes are compared to Pipe Reactor processing later in this paper.

Advantages of the Pipe Reactor Using Low Strength Acid

Acid strengths in the 38% to 43% P₂O₅ range are considered low strength when processing MAP. Dryer fuel is required and recycle rates are substantially higher than for acid strengths above 46% P₂O₅. However, the lower strength acids process very well at Texasgulf in the Pipe Reactor and because of the higher temperature of the slurry coming out of the Pipe (300°F to 330°F), more heat is injected into the granulator, resulting in more overall evaporation, more capacity, less recycle requirements, and less fuel for the Pipe than for a preneutralizer operation.

Low Citrate Insoluble P₂O₅ in MAP Product

Ammoniation to mole ratios of 1.0 or less in slurry vessels frequently results in high citrate insoluble losses, 0.5% or higher. Such losses are completely avoided in the Pipe.

A Low Cost Device

The Pipe Reactor itself is relatively nominal in cost for sizes up to 300mm (12"). Also, since emissions of ammonia and fluorine can be very low, scrubber modifications for conversions from TSP to MAP can be very low in cost. These factors make it ideal for a retrofit situation.

DISCUSSION

The Pipe Reactor for DAP

Over 300,000 tons of DAP were produced in 1986 and 1987 at the Royster plant at Mulberry, Florida using a 10" Pipe Reactor almost identical to the Pipe Reactor used by Texasgulf. The operation of the Royster plant has been rather well described in the 1986 article by Achorn and Paulson⁽³⁾.

The DAP operation at Royster was technically successful. However, the new management that took over the plant in May of 1987 apparently viewed the Pipe process as having no advantage over the conventional DAP process. The plant had a preneutralizer and a capacity potential in the conventional process above that which had been demonstrated using the Pipe.

This abandonment at Royster, probably the world's largest DAP pipe reactor plant while it operated, and abortive efforts at Pipe Reactor processing at Agrico, Farmland, Beker and almost all USA producers, has led to a reluctance by most USA manufacturers to consider a Pipe Reactor for DAP, or even MAP.

The USA consensus on Pipe Reactor processing of DAP is that the drier and hotter granulator conditions do not favor high rates of ammonia absorption. With high impurity acid like Florida, this puts an added burden on making grade.

Many of the advantages claimed for the Pipe Reactor on DAP have been achieved in the conventional process.

We can list the various factors that effect DAP process selection as follows in random order:

(1) Fuel Consumption

Many USA plants operate with very low or no fuel. The use of gaseous ammonia assists in achieving a higher temperature in the granulator. This approaches Pipe Reactor conditions, but also can increase ammonia slip and can reduce the extent of ammoniation.

(2) Demonstrated Capacity

The capacities achieved in USA plants are far in excess of any achieved in pipe reactor facilities to date. Most pipe reactors are small, some are supplemental units operating together with a preneutralizer. Demonstrated maximum capacities on DAP, using North African acid to date, are probably 65 MTPH. In the USA, one conventional DAP facility is currently running a stream rate of over 145 MTPH. There are probably about 10 single lines operating in the range of 90 MTPH of DAP. Therefore, there seems to be little incentive to modify to the Pipe Reactor mode for increased capacity.

(3) Recycle Rate

Pipe Reactor advocates claim reduced recycle requirements, under 4.0:1.0. Virtually all conventional DAP plants in the USA are already achieving this. One plant, currently making about 85 TPH DAP is achieving about 3.0:1.0 recycle or less, positively measured by a recycle scale. The use of gaseous ammonia in the granulator tends to reduce recycle requirements.

(4) Capital Cost

There is no evidence that equipment size or cost is higher using the conventional process. It is probably true that process suppliers using the conventional process would provide a comfortable safety factor in recycle. The plants capacity ultimately turns on the amount of recycle capacity available. Dryer sizes used in the conventional process in the USA are relatively small with high throughput a characteristic. It is probable that in any given DAP project, factors other than whether the plant employs a Pipe Reactor or not, are likely to account for any capital cost differences.

(5) Acid Quality

The acid used in Florida contains well known impurities in ever increasing amounts. Most plants must ammoniate to a 1.90 mole ratio of 7.5 to 7.6 pH, and even then urea is often necessary to reach 18.0% N. Plants using North African or Middle Eastern acid have so few impurities present that they frequently have trouble granulating and usually can make DAP at 7.0 to 7.3 pH.

We should also add that several producers in the USA believe that liquid ammonia is preferable for the granulator, from the standpoint of making product grade. They contend that the hotter, dryer conditions resulting from gaseous NH_3 are unfavorable. Since Pipe Reactor conditions are a further extension of these hotter, dryer conditions, these producers are the least likely of all to "Go Pipe".

Future projects, likely to employ high impurity acid, such as available in China, for example, should approach a DAP process using a Pipe Reactor cautiously.

(6) Startup Time

The conventional process is so well known that startup time can be a few days or less. Obviously, there is a substantial reservoir of operators familiar with the techniques as currently practiced in the plants mentioned above.

(7) Summary

At least 75% of the world's DAP production is made in USA plants using the conventional process. These plants have shown high productivity. The average facility probably operates on low grade acid at about 50% higher in capacity than the largest facility using a Pipe Reactor. While the U.S. industry may be accused of introversion, it is also highly competitive and not likely to overlook a technique that could provide a significant economic advantage.

If it can be demonstrated that a composite acid strength, significantly above 40% P_2O_5 , can be run on DAP using the Pipe Reactor, then increased output could be expected. So far, we have not seen any Pipe Reactor processing in which such higher acid strength has been used.

Even though the case for using a Pipe Reactor on DAP has the disadvantages listed above, there are situations where the Pipe Reactor should be considered for DAP.

Where the primary product of a TSP plant revamp, for example, is MAP, or where high quality acid is available, there can be situations in which the production of DAP using a pipe would be commercially attractive. This requires some forethought in the scrubber system revamp to cope with the higher ammonia slip resulting from DAP processing. However, in most cases, this should be perfectly feasible.

A generalized flowsheet for DAP production using the Pipe is shown in Figure 5. This illustrates the two-stage or dual mole scrubbing system which was also practiced in a slightly different form at Royster. While thorough, preferably two-stage, scrubbing is required to catch the ammonia in any DAP process, other scrubbing arrangements are possible with the Pipe, including arrangements using a single strength 38% to 40% P_2O_5 acid as the primary scrubbing medium.

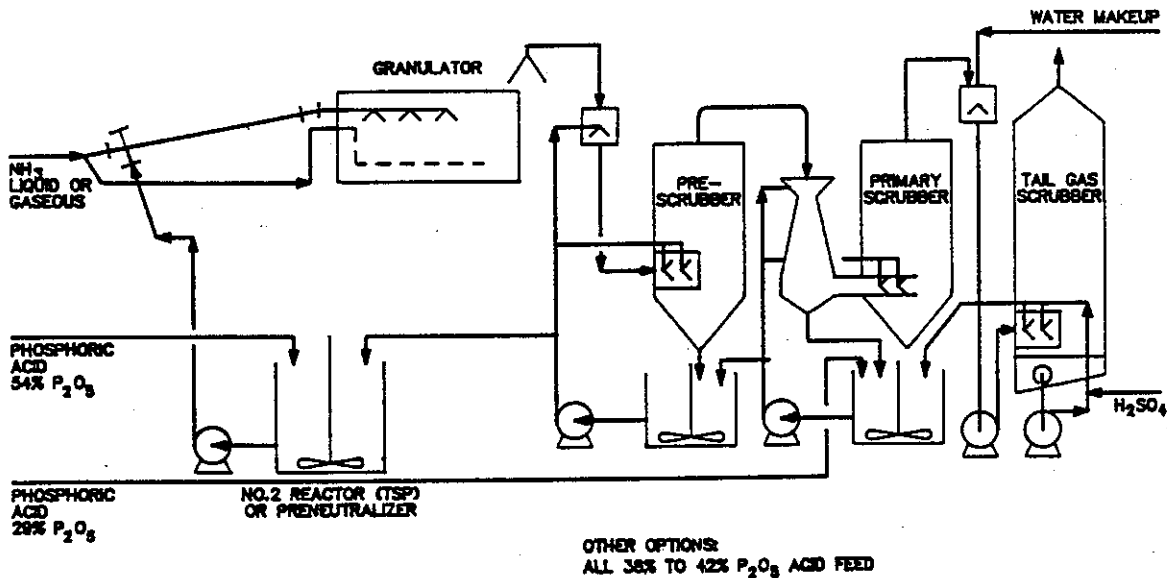


Fig. 5 GENERAL DAP PIPE REACTOR FLOWSHEET

Other MAP Processes

In the original Dorr MAP process, where all the neutralization was done in the slurry reactor or reactors, the low solubility of mono ammonium phosphate resulted in high moisture levels, high recycle, high fuel requirements, and low capacity. To avoid these problems, current producers use at least two other processes, besides the Pipe Reactor process. These processes are discussed below.

Granulator Ammoniation

In this process, originally operated, we believe by Agrico in the Dorrco drum granulator line at Faustina, Louisiana⁽⁴⁾, all the ammoniation is done in the granulator. No slurry ammoniation is done and the substantial ammonia slip is neutralized with high strength acid in the scrubbing system at a relatively low mole ratio to avoid high viscosity. This process has the advantage of being able to use higher strength acid than the "Back Titration" method described next, and therefore, operates at low recycle rates and high plant output. The process produces a very good quality granule.

Back Titration Method

This method is in practice in several plants. The preneutralizer is used, as in DAP, to produce a 1.4 to 1.5 mole ratio slurry using 38% to 40% P_2O_5 composite acid, some of which has gone through the scrubbing system at 29% P_2O_5 . In the granulator, 50% to 54% P_2O_5 acid is sprayed on the bed to back titrate the slurry to the desired product mole ratio. The composite acid strength is thus about 43% to 44% P_2O_5 , feeding 30% to 40% of the P_2O_5 as high strength acid.

This process utilizes, therefore, somewhat lower strength acid than can be used in either the Pipe Reactor process or the Granulator Ammoniation process. The ammonia slip is fairly low, coming mostly from the preneutralizer. Therefore, ammonia and fluorine emissions tend to be low. Some operators of the process contend that the product from the granulator tends to be sticky causing build-up in the dryer front end. The process avoids high citrate insoluble P_2O_5 .

Other Products by Jacobs' Pipe Reactor Processes

Sulfate Grades Like 16-20-0

In the original Dorr process for 16-20-0, where the mixed acids are ammoniated solely in the reactor(s), the low solubility resulted in a slurry at about 1.0 mole ratio of about 70% solids. This high level of moisture required high recycle and high fuel consumption. More recent processors have used several methods including Pipe Reactors to operate at lower moistures.

16-20-0 also has a very high heat of reaction so the preferred arrangement is to use both a Pipe Reactor and a preneutralizer. The sulfuric acid is fed primarily to the Pipe Reactor, up to its heat dissipation capacity of about 750,000 BTU/sq. inch of Pipe Reactor cross section area. The Pipe Reactor would need to be Hastelloy G-30 in this case. While some processes add H_2SO_4 directly to the granulator, Jacobs avoids this in order to protect the rubber lining.

High Nitrogen Grades Using Urea or Ammonium Nitrate

NP and NPK products made with ammonium nitrate or urea require special process design to limit the moisture in the granulation system.

When these grades, such as 15-15-15, or 17-17-17 are MAP based instead of DAP based, the use of the Pipe Reactor increases capacity by using a higher strength acid and injecting the entire reaction heat into the granulator. The Pipe also accommodates the addition of sulfuric acid to these grades to improve reaction heat and water evaporation. We believe it is best practice to use the Pipe Reactor for the sulfuric acid, avoiding the addition to the granulator where it can decompose urea or ammonium nitrate causing fume and submicron emissions.

Other Pipe Reactor Processes

ERT, CROS, CDF and Incitec Processes

The ERT and CROS processes from Spain, the CdF Chimie-AZF dual pipe reactor process of France, and the Incitec process of Australia have been rather well described in recent articles too numerous to mention. These processes are often applied to NPK products using slurries, usually in the 1.0 to 1.75 mole ratio range. In some cases, DAP is made, primarily using North African or relatively pure phosphoric acid.

Some trials and studies, using the above processes, have been made in USA plants for producing DAP, using Florida acid. However, at the present time, no plants in North America are using Pipe Reactors of the above processes for either DAP or MAP.

Pipe Reactor Processes Used in Blunger Plants

The Dorrco blunger-type of granulator presents a particular problem to the user of Pipe Reactors for large scale operation, because of the large volume of water vapor to be removed and the high velocity of the slurry exiting the pressurized Pipe. The freeboard in the blunger is relatively small compared to the space available in the granulator and fouling has resulted in abandonment of Pipe processing in some cases.

Two processes do use Pipe Reactors with blungers but both operate at relatively low rates of MAP or MAP/DAP mixtures, probably in the 20 MTPH to 35 MTPH range.

The Cominco process⁽⁵⁾, making 16-20-0 and 11-52-0, is used at Cominco and Sherritt Gordon, both in Canada. The Reactor utilizes a venturi mixing zone to achieve complete reaction without scaling.

The UKF process⁽⁶⁾ makes NPK's using ammonium phosphate, ammonium nitrate and potash, operating in the 1.0 to 1.5 mole ratio range. The plant has three lines of granulation using Dorrco blungers. The unusual feature of this process is the use of static mixers to effect ammoniation in a relatively short space.

SUMMARY

The Pipe Reactor offers advantages in making MAP and MAP-based NP and NPK fertilizers. These advantages need to be considered in spite of past failures and the disadvantages in attempting to make DAP in Pipe Reactors in the USA. An MAP process is available using a Pipe Reactor which can produce hard, low moisture, dust-free, granular MAP grades, from 10-50-0 to 11-55-0 depending on acid quality. The Pipe Reactor allows processing of MAP at higher acid strength than other processes and, therefore, at low recycle and high capacity. At the same time, the fluorine and ammonia emissions are well under Florida and EPA standards.

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