

# **FROM PHOSPHATE ROCK TO DAP AT LOWER COST**

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## SUMMARY

This article describes a hypothetical phosphate chemical complex in which the various plants coordinate to provide minimum capital and operating costs and simplicity of operation. The scope begins with receipt of phosphate rock and sulfuric acid and continues thru production of diammonium phosphate. This includes phosphate rock handling, phosphoric acid production and related cooling water, and DAP production.

### **HEMI PHOSPHORIC ACID PLANT**

The key element of this complex is use of the Hydro Hemi (hemihydrate) process for direct production of phosphoric acid at 41%  $P_2O_5$  concentration. For most scenarios the Hemi process uses phosphate rock as received - without drying or grinding. Two entire plant sections are eliminated - rock grinding and phos acid evaporation. Cooling water, acid storage, clarification, and steam distribution systems are reduced to a small fraction of their conventional size. Capital cost for the phosphate complex is roughly 20-25% less than for a dihydrate-based complex, which would have required rock grinding, evaporation, larger cooling water and steam distribution systems, and often elaborate clarification systems.

### **HIGHER OVERALL $P_2O_5$ RECOVERY**

Overall phosphate recovery tends to be better with a Hemi process than with a dihydrate process - even when there is no advantage in recovery as

measured in the filter cake. The advantage is further enhanced where gypsum pond water is recirculated to wash the filter cake. Sulfuric acid requirement is about 5% lower than in a dihydrate plant, thanks to a combination of high recovery and operation at a low free sulfate level.

## **STABLE, EASY OPERATION**

Modern Hemi phosphoric acid plants tend to be easier to operate and require less cleaning than dihydrate plants. One reason for this is that the reaction takes place in a stable range of hemihydrate crystals, whereas dihydrate plants must (out of economic necessity) operate near the unstable transition between dihydrate and hemihydrate.

## **DAP PLANT FEATURES**

The DAP plant features facilities which provide excellent ammonia recovery, vaporization of all ammonia with free energy, low atmospheric emissions, enhanced screening efficiency, and a simple enhancement for product cooling. Hemi phosphoric acid is purer than dihydrate phosphoric acid, providing capability to make DAP with 2% higher  $P_2O_5$  content.

## **ENERGY EFFICIENCY**

This complex provides major energy efficiency benefits. It includes typical sulfuric acid plants and co-generation of steam and electric power from heat of burning sulfur. No process steam is required for the Hemi phosphoric acid plant or the energy-efficient DAP plant. No power is required for grinding rock or concentrating phosphoric acid. Therefore, there will be much more surplus electric power than would be available from a chemical complex using a dihydrate phosphoric acid plant. This 1500 metric T/D  $P_2O_5$  complex not only provides all of its own energy requirements from process heat, it is also a major exporter of electric power. Total power generation is near 60 megawatts - worth about \$20 million annually. Energy efficiency associated with the Hemi phosphoric acid plant account for about 30% of this power production.

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## TABLE OF CONTENTS

	<u>Page</u>
<b>SUMMARY</b>	<b>1</b>
<b>TABLE OF CONTENTS</b>	<b>3</b>
<b>PHOSPHATE ROCK HANDLING</b>	<b>4</b>
<b>THE HEMI PHOSPHORIC ACID PROCESS</b>	<b>5</b>
Acid Concentration	5
No Evaporation or Steam Required	6
Acid Storage & Clarification	6
Cooling Water Requirement	6
Raw Material Consumption	7
Operating Stability	8
Gypsum Crystallization Graph	9
Processing Impure Rock	10
The Hemi-Di Process Option	10
Hydro Hemi & Hemi-Di Process Experience	11
<b>THE DAP PLANT</b>	<b>13</b>
BFL Vaporizer-Scrubber	13
Double-Mole Reactor/Granulator Scrubbing	14
Reactor Design	15
Product Screen Diverter System	15
Feed Acid Concentration	16
Tran-Tech Product Cooler	16
<b>ENERGY UTILIZATION</b>	<b>17</b>
<b>REFERENCES</b>	<b>18</b>
<b>ABOUT THE AUTHOR</b>	<b>19</b>

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## PHOSPHATE ROCK HANDLING

The Hydro Hemi phosphoric acid process can use rock which is much coarser than that required for conventional dihydrate processes, so grinding is not required for most of the world's phosphate rock sources. Particle size requirement for Hemi is typically -2 mm (9 mesh), compared to a requirement of -35 mesh for typical dihydrate processes. The Hemi process can handle damp rock with up to about 15-20% moisture. Most commercial phosphate rock sources worldwide are suitable in particle size and moisture content for feeding directly to a Hemi plant without drying or grinding. This includes coarse concentrate and some screened phosphate rocks.

An important exception is that the pebble rock which makes up a significant (but decreasing) portion of central Florida phosphate is much too coarse. Pebble rock could be ground to -2mm size in relatively low energy impact, hammer, or roller mills, with closed circuit screening. This pebble rock is available dripping wet with about 10% moisture, and drying would be quite expensive. Consequently, the rock would be ground without drying, followed by wet screening and recycle of damp +2 mm material to the mill. Such milling requires only a fraction of the power and capital cost that a ball mill requires. Rod milling of the damp pebble would be acceptable, but probably unnecessarily expensive in capital and power cost.

Ball milling of rock for the Hemi process is unnecessary. Conventional wet ball milling would be unacceptable, because wet ball mill slurry containing about 38% water is too wet for the Hemi plant water balance.

## THE HEMI PHOSPHORIC ACID PROCESS

The most widely used Hemi process was developed by Fisons in the 1960's and is now licensed by Hydro Fertilizer Technology. As in nearly all commercial phosphoric acid plants, phosphate rock reacts with sulfuric acid to produce phosphoric acid and gypsum crystals. The Hemi process operates at a high concentration of phosphoric acid, where the gypsum crystals exist as the hemihydrate form of calcium sulfate ( $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ ). The alternative dihydrate process operates at a much lower concentration of phosphoric acid, where the gypsum crystals exist as the di-hydrate form of calcium sulfate ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ).

### **ACID CONCENTRATION**

Hemi plants can produce phosphoric acid directly from filtration at concentrations ranging from 38% to 50%  $\text{P}_2\text{O}_5$ , but optimum is usually around 41%. Most dihydrate phos acid plants make 25-28%  $\text{P}_2\text{O}_5$  product. Hemi acid concentration is more than adequate for DAP feed acid, as was clearly demonstrated at Belledune, Canada, where phos acid evaporators and steam boilers were never needed. The Belledune DAP plant received phos acid at single strength for both scrubbing and reactor feed purposes.

To meet the DAP plant requirement of 38%  $\text{P}_2\text{O}_5$  average phos acid concentration, the Hemi plant here would provide about 70% of its acid as 41%  $\text{P}_2\text{O}_5$  filter product, which would be routed to the DAP reactor. The remaining 30% of the phos acid would be withdrawn as #2 filtrate at 30-35%  $\text{P}_2\text{O}_5$  and used for scrubbing at the DAP plant.

## **NO EVAPORATION OR STEAM REQUIRED**

No phosphoric acid evaporators are required in this scenario, and no steam is used in the process. If alternative products such as MAP or TSP were produced, some evaporation would be required, but it would retain the same concentration and energy efficiency benefits relative to a dihydrate plant.

## **ACID STORAGE AND CLARIFICATION**

Phosphoric acid storage and clarification facilities are reduced to about one third that required for a dihydrate plant. There will be agitated storage tanks for 30% and 41% acid. Clarification is not necessary because the 30% and 41% phosphoric acids have both come from filtration, and because the high purity of Hemi acid makes further clarification unnecessary.

A dihydrate plant would typically require storage and clarification for 26% and 40% acid, plus agitated storage for 52-54% acid.

## **COOLING WATER REQUIREMENT**

There is no need for the large flow of cooling water normally required by phosphoric acid evaporators. A relatively small flow of cooling water is required for flash cooler condensers, fume scrubbing, equipment washing, etc. This water does not need to be as cool as the 33-35°C (92-95°F) required for 52-54% P<sub>2</sub>O<sub>5</sub> evaporators in a dihydrate phosphoric acid plant. Water at around 38°C (100°F) will be adequate for scrubber water, equipment wash, etc., and more than adequate for flash cooler condenser water. The cooling pond and associated pumps and piping are a fraction the size of comparable equipment in a dihydrate plant.

## RAW MATERIAL CONSUMPTION

Recovery of  $P_2O_5$  as measured in the filter cake will be roughly equal for the Hemi or dihydrate process (varying considerably with type of rock, condition of the plant, and how hard the plant is pushed for capacity). The Hemi process benefits from less loss of product elsewhere in the plant, which should amount to at least 1% better recovery. This comes from elimination of any losses relating to handling of 26%  $P_2O_5$  acid or from evaporation.

There is an added recovery benefit for plants which recirculate gypsum pond water. Dicalcium phosphate which crystalizes within the gypsum crystals will re-dissolve in the recirculated pond water. Much of this re-dissolved phosphate is recovered when the same pond water is used to wash the filter cake. This amount to about 2% recovery benefit. Overall  $P_2O_5$  recovery in a Hemi plant will thus be about 2.5% better than in a dihydrate plant, based on equal filter cake loss for both plants, as summarized below:

<b><math>P_2O_5</math> Recovery Example, Hemi vs. Dihydrate</b>		
	<b>HEMI</b>	<b>DIHYDRATE</b>
<b>FILTER RECOVERY</b> (Based on filter cake)	<b>95%</b> (3.5% CS loss, 1.5% WS loss minor Cl loss)	<b>95%</b> (3.5% CS loss, 1.5% WS loss minor Cl loss)
<b>26% Acid Handling &amp; Evaporation Losses</b>	<b>0</b>	<b>-1%</b>
<b>Miscellaneous Losses:</b> (From filter pans or belt grooves, flash cooler entrainment, spills, leaks, and other acid handling)	<b>-2%</b>	<b>-2%</b>
<b>Recovery from pond water wash of filter cake:</b> (@50% recovery) <b>Re-dissolved dicalcium phosphate (C.S.):</b> <b>Water soluble (W.S.) losses:</b>	<b>+2%</b> <b>+1.5%</b>	<b>0</b> <b>+2%</b>
<b>Overall <math>P_2O_5</math> Recovery</b>	<b>96.5%</b>	<b>94%</b>



Sulfuric acid requirement for a Hemi plant benefits from the fact that the ratio of free sulfate to  $P_2O_5$  in a hemi plant is substantially less than in a dihydrate plant. Another small benefit is that there is less sulfuric acid reacting with aluminum impurities in the phosphate rock. The net outcome of these two effects is about 2.5% Hemi benefit. Otherwise, sulfuric acid consumption will be inversely proportional to  $P_2O_5$  recovery. Thus if a Hemi plant has 2.5% better overall  $P_2O_5$  recovery listed in the preceding table, plus the 2.5% sulfuric acid reaction benefits, it will consume 5% less sulfuric acid than the dihydrate plant.

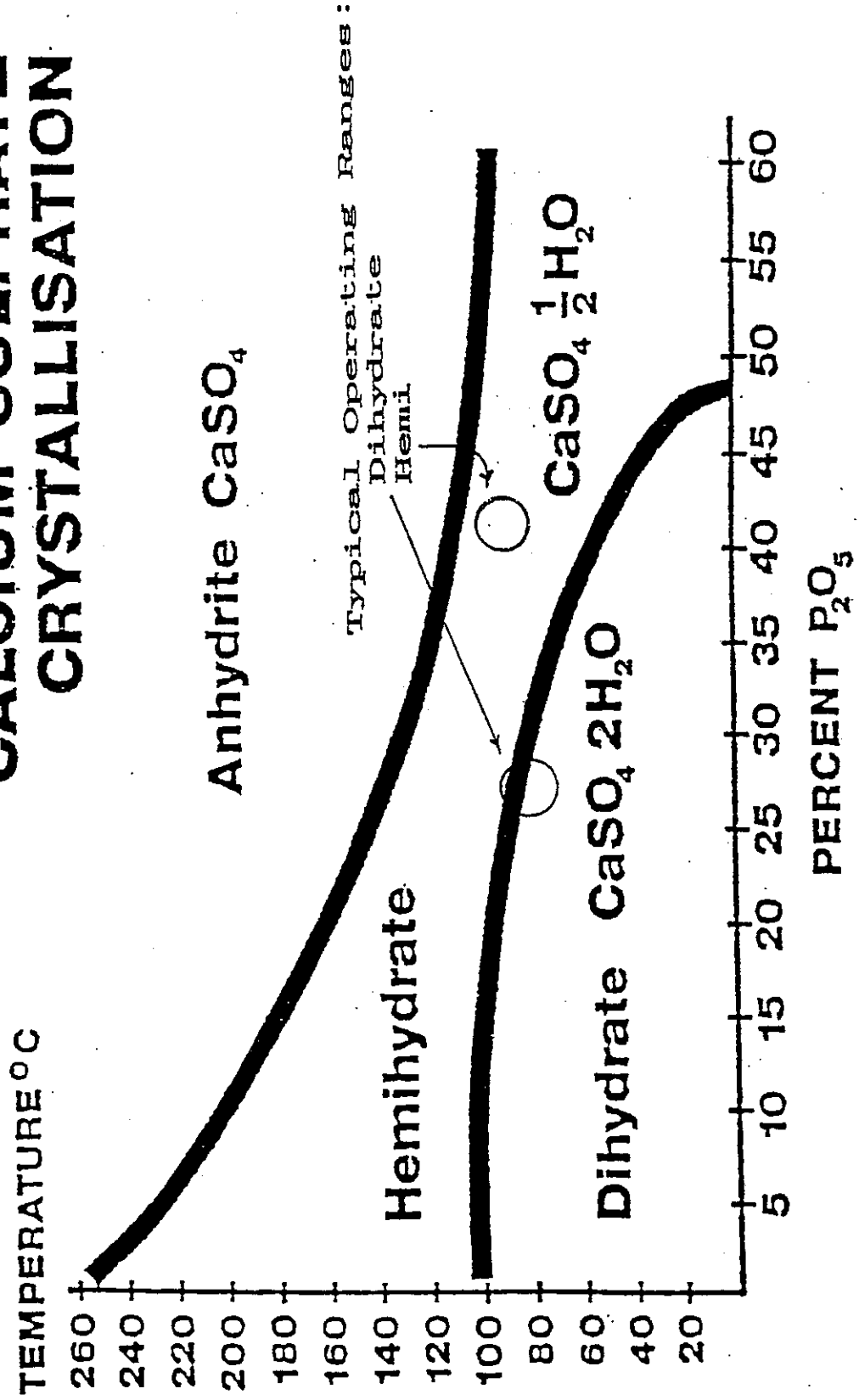
### **OPERATING STABILITY**

Either the hemihydrate or dihydrate process can operate stably if the conditions are clearly in either the hemihydrate or dihydrate zone. Hemi plants operate in a stable zone, which accounts for praise by those that operate the plants that they are easier to operate and more tolerant of upset conditions than dihydrate plants. There is relatively little scale formation in the reactors and flash coolers, because of operating in the stable zone, and because there is lower solubility of calcium sulfate.

For economic reasons dihydrate plants must push into the transition zone between hemihydrate and dihydrate. This is illustrated in the calcium sulfate crystallization graph on the following page. The crystals in a typical dihydrate plant are a mixture of some hemihydrate among mostly dihydrate crystals. Dihydrate plants tend to have much more scale formation in the reaction and flash cooling systems.

In a Hemi process conditions in filtration pass thru the Hemihydrate/dihydrate transition line. Technology has been developed which allows this transition to be crossed with minimal scaling in the filter system. Part of this technology has been use of an anti-scalant reagent, which greatly slows the conversion of hemihydrate crystals to dihydrate crystals, thus reducing scaling. Anti-scalant is not always necessary, as demonstrated at Belledune, where a modified Bird tilting pan filter was operated most of the time with no anti-scalant.

# EFFECT OF REACTION CONDITIONS ON CALCIUM SULPHATE CRYSTALLISATION



## **PROCESSING IMPURE ROCK**

There is increasing evidence that the Hemi process performs well with some types of phosphate rock which are impractical to process with conventional dihydrate processes. This includes rock with very low  $P_2O_5$  concentrations and unusually high levels of iron, aluminum, and magnesium impurities. Such rock is either left in the ground, blended with higher grade rock, or processed for further purification. Ability to process such rock could lead to breakthroughs in utilizing phosphate reserves in places like South Florida.

## **THE HEMI-DI PROCESS OPTION**

A variant of the Hemi process is the Hemi-Di process, which employs a second reaction section followed by a second filtration section to achieve extremely high recovery of 98-99%. The Hemi-Di process minimizes raw material costs, produces cleaner gypsum, has cleaner cooling pond water, can accept more cake wash water, and in special circumstances it provides an opportunity to recover sulfuric acid from certain weak acid solutions.

## HYDRO HEMI & HEMI-DI PROCESS EXPERIENCE

The Hydro Hemi process has been chosen since 1970 for 10 Hemi plants and 10 Hemi-Di plants which have been built, converted, or are underway worldwide. These are listed in the following two tables. HiTech Solutions Inc. is North American licensee for the Hydro processes, and offers design of Hydro Hemi and Hemi-Di plants for most other sites worldwide.

HYDRO HEMI PLANTS					
CLIENT	Location	Design t/d P <sub>2</sub> O <sub>5</sub>	Actual mt/d P <sub>2</sub> O <sub>5</sub>	Year	ROCK
Windmill* **	Holland	610		1970	C.Fla., Togo, Moroc.
HCI	Cyprus	130		1982	Algeria, Jordan, Togo
Windmill* **	Holland	330		1983	C.Fla., Togo, Moroc.
Royster	Fla., USA	590	650	1985	C. Fla. Spiral conc.
Belledune	Canada	500	600	1986	C. Florida
Arcadian*	La., USA	655	800	1990	BuCraa
Serrana*	Brazil	480	580	1994	Brazil
Indo-Jordan*	Jordan	700	870	1997	Jordan
Western Mining	Australia	1500	construction	1999	Queensland
Hydro-Agri Jordan	Jordan	1420	Bidding	2001	Jordan

\* Operating \*\* Two Windmill Hemi plants were converted into one Hemi-Di plant in 1991.

HYDRO HEMI-DI PLANTS				
CLIENT	LOCATION	metric t/d P <sub>2</sub> O <sub>5</sub>	YEAR	ROCK
RMKH Trepka*	Yugoslavia	160	1974	Jordan
Albright & Wilson	England	500	1980	Morocco
CSBP	Australia	500	1981	C. Florida
Pivot	Australia	100	1981	C. Florida
Supra***	Sweden	360	1986	C. Fla, Morocco, Togo
Chinhae*	South Korea	250	1990	C. Florida, China
Hydro-Agri* **	Holland	620	1991	C. Florida, Morocco
Hong He Zhou*	China	210	1993	Jian Chuan China
NFC*	Thailand	700	1997	Jordan, Morocco
Sterlite***	India	420	1999	Jordan

\* Operating

\*\* Two Windmill Hemi plants were converted into one Hemi-Di plant in 1991.

\*\*\* The Supra plant was relocated to Sterlite in India. Now ready to start.

The writer was responsible for process engineering for the two most recent conversions of North American phosphoric acid plant to the Hydro Hemi process - Belledune Fertilizer in Belledune, New Brunswick, Canada in 1986; and Arcadian Corp. in Geismar, Louisiana in 1990. These plant achieved design performance in 2-19 days after startup, soon demonstrated 110-120% of design capacity at 95-96% filter cake recovery, and were praised by operating personnel as being exceptionally easy to run. References and visits to Hemi plants can be arranged to demonstrate experience of users of the Hemi process.

Further information on the Hemi and Hemi-Di processes and operating experience at several plants can be found in references cited at the end of this article, including papers presented at Clearwater in 1991 and 1995.

## THE DAP PLANT

Granular diammonium phosphate is produced in a single plant with capacity of 135 metric tons per hour. Costs of capital, ammonia, steam, and operation are minimized by incorporating several innovations:

- All ammonia feed is vaporized with free heat from the DAP process in a patented **BFL vaporizer-scrubber**, eliminating the cost of steam heat.
- This ammonia vaporizer doubles as an efficient tail-gas scrubber, cleaning and cooling the exit air.
- Fumes from the granulator and reactor (pre-neutralizer) are scrubbed with acid in **two stages of counter-current scrubbing** - providing near zero loss of ammonia.
- The combination of 2-stage acid scrubbing, the BFL vaporizer-scrubber, and efficient cyclone and scrubber design **eliminates the need for a separate tailgas scrubber**.
- The **DAP reactor** is large at the top to minimize entrainment, and small at the bottom to minimize formation of an insoluble phosphate compound.
- **Screening** equipment is minimized with a system which routes only enough material as is required to the product screens.
- A simple **Tran-Tech Cooler** is employed to supplement cooling of the product entering the storage building.

### **BFL VAPORIZER-SCRUBBER**

This system uses waste heat from the reactor-granulator acid scrubbing system to vaporize all ammonia feed a DAP or MAP plant. "BFL" refers to Belledune Fertilizers Ltd., who developed and patented the process. It is licensed internationally by HiTech Solutions Inc. of Lakeland, Florida, USA.

The unit is located in the gas stream exit the reactor-granulator acid scrubber. Condensate from the hot gas side is separated and recirculated as scrubbing water. This condensate contains very low concentrations of ammonia, fluoride, and phosphate, so surplus condensate can be discharged into recirculated pond water or other impure water stream.

Vent gasses from the dryer, cooler, and equipment vents contain lesser amounts of contaminants. With efficient cyclones and well-designed acid scrubbers, the system will meet rigorous air emission standards, such as those of the US EPA. No separate tailgas scrubber is required with this system.

An environmental benefit of using phosphoric acid from Hemi plant is that it tends to contain about one third less fluoride than acid from dihydrate plants, evaporated to similar concentration. At the Hemi plant in Belledune, Canada, it was observed that this reduction in fluoride content of the phosphoric acid resulted in lower emission of fluoride from the DAP plant stack.

An alternative location for the BFL vaporizer is in the gas stream from the reactor. Advantages of the alternative location are that the vaporizer can be smaller, and the acid scrubbing system runs considerably cooler. Disadvantages are that it does not provide any tailgas scrubbing, and it produces condensate with a large amount of ammonia, which must be re-consumed into the process.

### **DOUBLE-MOLE REACTOR/GRANULATOR SCRUBBER**

Most of the ammonia and fluoride fumes in a DAP plant originate at the reactor and granulator. These fumes are cleaned with 2-stage counter-current acid scrubbing, controlled at two different N/P mole ratios. Advantages of this "Double Mole Scrubbing" are:

- Ammonia losses are greatly reduced.
- It is much easier for the main acid scrubbing system to operate at optimum mole ratio, because it is protected from excess ammonia.
- A tendency to emit sub-micron ammonium fluoride mist is avoided.
- When this is followed with a BFL Vaporizer-Scrubber, exit air is so clean that no tailgas scrubber is needed.

The first stage of Double Mole Scrubbing is controlled at an N/P mole ratio which is well above 1.0, and the second stage is done at well under 1.0 mole ratio. Mole ratio near 1.0 is always avoided, because that is a minimum solubility point, where severe scaling and fouling occur.

This 2-stage counter-current scrubbing system is highly effective in removing the two serious contaminants in this gas stream - ammonia and fluorides. Ammonia loss will be well under 1%, versus typically 2-5% for single-stage scrubbing. Double Mole Scrubbing is also more tolerant of high ammonia emissions from the reactor and granulator - a situation which otherwise would cause serious fouling problems by driving the mole ratio to 1.0.

Double Mole Scrubbing should be standard equipment in all new DAP plants, and it is frequently an attractive modification for existing plants, depending on actual ammonia losses and its value.

## **REACTOR DESIGN**

The reactor (pre-neutralizer) where phosphoric acid slurry is ammoniated has a large upper diameter to reduce entrainment of slurry to the vent duct. This large diameter would be detrimental if it increased the slurry residence time, because of a side reaction which renders some of the phosphate insoluble. Therefore the lower part of the reactor is smaller in diameter, to limit slurry residence time and minimize the undesirable reaction. This innovation was pioneered by the owners of HiTech Solutions in designing the W.R. Grace (now Cargill) #4 DAP plant.

## **PRODUCT SCREEN DIVERTER SYSTEM**

An innovation allows use of fewer product screens to achieve separation of fines and on-size material. In conventional granulation plants, these screens receive all recycling material after the coarse screens have removed oversize. In this plant a system of diverters and by-pass chutes limits the product screen feed to no more feed than is required to provide enough product. This minimizes the number of product screens that are needed, and improves product size distribution.



## **DAP FEED ACID CONCENTRATION**

Feed acid averaging approximately 38%  $P_2O_5$  is required, as is typical for modern DAP plants. Hemi phosphoric acid plants usually operate best at a slightly higher concentration. One option would be to receive all phosphoric acid at a single strength around 40-41%  $P_2O_5$  and to dilute with water as required to average 38%  $P_2O_5$ .

The preferred option is to use a small stream of #2 filtrate from the phosphoric acid filter at 30-35%  $P_2O_5$  for DAP scrubber acid. The main stream of 41%  $P_2O_5$  would be routed directly to the DAP reactor. This option helps phosphoric acid recovery by allowing a little more filter cake wash water. It helps DAP scrubbing efficiency by using acid which contains less fluoride and  $P_2O_5$ .

## **TRAN-TECH PRODUCT COOLER**

The main product cooler is a conventional rotary drum cooler of modest size. A simple machine combined with special procedures supplements cooling of the product in the storage building. A mechanical device provides enhanced cooling with ambient air, and the product is then distributed over an extended area. This supplemental cooling system is less expensive than alternatives such as using an ammonia cooler-chiller or increasing the size and air flow through the conventional cooling system.

The Tran-Tech Cooler was invented and patented by Mr. Sa Cao Tran and is licensed worldwide by HiTech Solutions. It is described in a paper by Sa Cao Tran and James W. Cox at this same 1999 Clearwater Convention.

## ENERGY UTILIZATION

The energy source of a phosphate chemical complex is sulfuric acid production. Surplus heat from burning of sulfur is absorbed by steam, which is used to generate all electric power required by the complex plus an export of electric power.

Energy efficiency of this phosphate complex is greatly enhanced by use of the Hemi phosphoric acid process. When the final product is DAP, no steam heating is required to concentrate the phosphoric acid, because the acid as produced is more than strong enough. A 1,500 T/D  $P_2O_5$  phosphoric acid plant will save about 2500 T/D in evaporator steam by making filtered acid which is strong enough for DAP feed, compared to dihydrate process acid at 26%  $P_2O_5$ . The DAP plant uses no steam to vaporize ammonia, because the BFL vaporizer-scrubber does it with waste heat from the process. Over 3 megawatts of electric power is saved by not having to grind most types of rock, and by not having a phosphoric acid evaporation section.

This surplus steam would typically be used to generate electric power. Total electric power production is near 60 megawatts for a 1500 metric T/D  $P_2O_5$  phosphate complex - worth over \$20 million annually (based on 4 cents/kwh). The energy advantages associated with the hemihydrate process and BFL vaporizer account for about 18 megawatts of this power - worth \$6 million/year, or \$12/ton of  $P_2O_5$ .

The surplus electric power could be exported to the power grid for sale, or it could be wheeled to the owner's mine. If exported power is sold, it is crucial to negotiate a fair price.

It is important to note that any utilization of energy from waste heat is environmentally friendly. This electric power is produced with incremental net results of no pollution, no solid waste, and no consumption of fuel. No other source of energy can top this for ecological responsibility - whether it uses coal, oil, gas, nuclear fuel, wind, or solar energy.

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# HiTECH SOLUTIONS, Inc.

ENGINEERING DESIGN CONSULTING

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## ABOUT THE AUTHOR

**John H. Wing** - Vice President of HiTech Solutions, Inc. - has a Bachelor of Chemical Engineering with Honors from Univ. of Florida and a Master of Engineering in Administration from Univ. South Florida. He has served the phosphate industry for over three decades in process design, consulting, project management, consulting, technical service, process development, and production supervision.

He has designed modifications and expansions of several phosphoric acid, DAP, MAP, and GTSP plants. He performed the process design for fluosilicic acid recovery systems at Conserv in Florida and Sterlite in India. He provided conceptual design for five phosphoric acid evaporators and about fifty scrubbers.

For Hemi plants he performed the process design for the Belledune and Arcadian Hemi conversions, was also project manager for the Arcadian Hemi conversion, and consulted as owner's engineer for the Indo-Jordan Chemical Co. Hemi plant in Jordan. He has written 9 technical papers on the Hemi and Hemi-Di processes, phos acid evaporation, and cooling pond systems, and has contributed several article segments to *Phosphorus and Potassium* magazine.

He is a registered Professional Engineer, Fellow of AIChE, and past Chairman of the Central Florida AIChE Section.

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