START-UP OF OXY'S NEW ANIMAL FEED GRADE DICAL PLANT

BY
JORGE E. SIERRA

Introduction

This paper will describe the construction and start up of the animal feed grade Dicalcium Phosphate facility of Occidental Chemical Company at White Springs, Florida. The chemical process will be reviewed, without proprietary details. This will be followed by a description of the construction chronological sequence. Then the start up and operation of the facilities will be exposed without revealing proprietary details.

Review Of The Process

1) Granular Dical

The process to make Dical is similar to any granular phosphate fertilizer process. It is particularly similar to the TSP process.

To make TSP, phosphoric acid is reacted with phosphate rock in the presence of water and steam. The resultant slurry is sprayed on the dry granular recycle and then dryed and screened.

To make Dical the phosphoric acid is first defluorinated, then it is reacted with limestone in the presence of water.

Two grades of Dical are produced by OXY in White Springs, and in Davenport, Iowa: One contains 21.00% P and 15.00% Ca minimum. The other contains 18.5% P and 19.00% Ca minimum. The bulk density of both of these two grades is typically 56.00 lb. per cubic foot. The process for each grade is slightly different. The 21% P grade requires the reaction to be completed more than the 18.5% P grade.

Excellent granulation is easily achieved when making 21% P grade. But good granulation is more difficult when 18.5% P grade is made. The reason is that the 21% grade requires a higher proportion of acid. Some of this acid ends up unreacted, and available for granulation.

The 18.5% P grade requires so much limestone, that all the acid is reacted and none is left to granulate. It is OXY's proprietary process which surpasses the difficulty of overgranulation or undergranulation whichever the case may be. OXY's process is such that good granulation is obtained in the 18.5% P grade manufacturing, and in the 21.0% P grade the granulation is suppressed by guaranteeing a complete reaction before the slurry is sprayed on the recyle.

The end product in both cases is a hard, nonsticking granule which results in long shelf life and very little attrition when handled in shipping and unloaded by the customer.

The reaction to make Dical takes place according to the following equation:

$$2H_3PO_4 + CaCO_3 \longrightarrow Ca(H_2PO_4)_2 \cdot H_2O + CO_2$$

For the 21.0% P grade and:

$$H_3PO_4 + CaCO_3 \longrightarrow CaHPO_4 + CO_2 + H_2O$$

For the 18.5% P grade.

The resulting slurry is sprayed on the dry granular recycle as in TSP then dryed and screened with double deck hummer screen machines. The limestone is 98% $CaCO_3$ and the acid has a P to F ratio of 100 or larger. The 100 to 1 ratio is the minimum required for animal feed. Typical ratios in the OXY Dical is 120 to 1. For example: 21.3% P, 0.18% F.

The acid used for fertilizer is not defluorinated. In TSP production, a portion of the P_2O_5 in the product is supplied by the phosphate rock while all the P_2O_5 in Dical comes from the acid.

2) Acid Defluorination

In simple form the reaction that takes place in the acid defluorination process is as follows:

$$4\ HF + SiO_2 \longrightarrow SiF_4 + 2\ H_2O$$

The SiO_2 is in the form of Diatomacous Earth (D.E.), which is 70% SiO_2.

The SiF_4 is stripped from the acid by air, sparged at the bottom of the coned bottom tank, and pulled to the scrubber by the scrubber fan. These fumes tend to condense on the conveying ducts' walls if the temperature falls below the dew point.

The fumes are scrubbed in a horizontal cross flow scrubber which uses Polypropylene honeycomb type packings. A pressure booster pump maintains adequate pressure in the fog nozzles. The incoming pond water is trained upstream of the pump to keep the spray nozzles from plugging.

The condensed fumes, in the form of fluosilisic acid, are carried with the scrubbing water and pumped back to the central cooling pond.

The scrubbing reaction is as follows:

$$3\ SiF_4 + 2\ H_2O \longrightarrow 2\ H_2SiF_6 + SiO_2$$

The quantity of the scrubbing water must be sufficient to wash the precipitated silica in the scrubber and on the packing. This is guaranteed by maintaining approximately 50 psig pressure on the spray nozzles when they are clean.

Construction Of The Facilities

The construction was done in three phases:

Phase I: The existing granulation train was converted to produce Dical.
Phase II: The storage and shipping facilities were built.
Phase III: The Phosphoric Acid Defluorination unit was built.

The entire project took 18 months for completion.

In order to put the project in perspective, it is desirable at this point to describe the complex in which the Dical plant was built. Then the circumstances leading to the decision to make Dical at White Springs will be delineated. Following that, the modification to the existing plant and the new construction will be described.

Suwannee River Chemical Complex Overview, and Factors Leading To The Conversion

1) Overview

OXY's White Springs Operation is composed of two chemical complexes and two phosphate benefication plants. The Suwannee River Chemical Complex (SRCC) and the benefication facility serving it were built in 1965-1966.

The SRCC is composed of the following facilities:

1. Four (4) sulfuric acid trains with total capacity of 5,400 TPD of 93% H_2SO_4.

2. Three (3) phosphoric acid trains (four filters) with a total capacity of 2,000 TPD of P_2O_5.

3. Eight (8) secondary evaporators capable of concentrating the 2,000 TPD of P_2O_5 produced by the filters. The acid is concentrated to 40% and 54% P_2O_5.

4. Twelve purification filters to prepare the merchant acid for the production of SPA.

5. Two (2) SPA producing trains.

6. Two (2) ball mills to grind the phosphate rock for the production of fertilizer and one of the animal feed plants (Pollyphos).

7. Two (2) granular fertilizer plants capable of producing 1,400 TPD of DAP and 700 TPD of triple super phosphate.

8. Two (2) animal feed grade phosphate plants each with its own shipping facilities:

a. Pollyphos

b. Dical

This is the complex where the Dical facility is located.

2) Background Leading To The Conversion

In 1975 OXY expanded it's DAP production by building a new plant (Z-Train) to make 1,400 TPD of DAP.

Up to that point in time the fertilizer was produced in two identical granulation trains each capable of making 600 TPD of DAP or TSP (Diamonium Phosphate or Triple Super Phosphate). These two trains (X and Y), mirror image of each other, were built in 1965.

When the TSP market softened, Y-Train was put to produce DAP and X-Train was converted to produce Dicalcium Phosphate to be marketed as an animal feed mineral supplement.

Modification Of The Converted Plant

Since the process to make Dical is very similar to the granular TSP process, not many changes in equipment were necessary to make the conversion. Some of the work involved washing the entire plant, and putting the equipment in operable condition.

Some of the equipment added or removed cannot be discussed here due to the proprietary nature of the process. But in general, equipment was added or modified, to guarantee a complete reaction (for the production of the 21% P grade) and to reduce the size of the recycled oversize. Some pollution abatment equipment was removed from the process since the phosphoric acid fed to the process has very low fluorine content. The fluorine emissions and the particulates are the only possible pollutants from the process.

The New Construction

1) Storage Silos

The existing storage buildings used for fertilizers were ruled out for Dical storage due to the high risk of contamination with the high fluorine fertilizer. It was decided, therefore, to erect enclosed silos. The closest real estate available for the silos was approximately 300' from the plant. Therefore, a conveying system was installed from the plant to the silos. Seven (7) belt conveyors were installed to transport the product to the top of the silos, and then to distribute it into each of the five (5) silos. Reversing conveyors were chosen instead of a tripper type conveyor, since, due to the position of the silos, the tripper belt had to

be reversible. The longest conveyor is approximately 300' long. It is supported at four points. The tail end is at an elevation 50' from the ground. The drive end is 110' above the ground and 20' above the top of the silos. Five silos were built, each one with capacity to store 2,000 tons. All the silos have cone bottom. A vibrating dish was fitted to the end of the cone, with isolation rods, and a flexible connection. The vibrating dishes have a "hat" in the center, inside the dish, which vibrates with the dish and deflects the material from the center toward the walls of the dish. This action is transmitted to the material in the silo and produces a very uniform discharge and avoids rat tail bridging.

Pan feeders were installed to feed the product from the silo to a conveyor which runs under the five (5) silos.

2) The New Shipping Facility

This part of the project was designed to load product in bulk and in bags.

A belt conveyor under the discharge of all five (5) silos discharges into a bucket elevator.

The product is passed thru a Rotex screen which removes any lumps bigger than 0.5". Then the product is sprayed and mixed with mineral oil for dedusting.

A loading bin, with capacity for 100 tons of product was built above two RR tracks. This gives the capability of loading on two tracks simultaneously, either on RR hopper cars or in bulk trucks or both.

A weight scale in the loading belt tells the loader the amount loaded in the vessel or going into the loading bin.

The product is bagged in closed, filling valve bags with a four spouts bagging machine. Each spout can fill one 50# bag in 15 seconds. This gives a bagging rate of 24 tons per hour.

The bags are conveyed to the vessel being loaded by a flexible spring conveyor. This conveyor is 80' long. OXY ships product in 50#, 55# and 110# bags. The 50# bags being for a domestic customer and the others for export sales.

Typical inventory levels are: 3,000 tons of the 18.5% P grade, and 5,000 tons of the 21.0% P grade.

Jacksonville is the port used most of the time for bulk and bagged material. But Tampa and Miami are also used.

3) The New Acid Defluorination Unit

The wet process phosphoric acid used to make the OXY Dical is stripped of its fluorine in this unit. It consists of cone bottom tanks with air sparging header, circulating pump, and heat exchanger.

A storage tank was built to hold 1,000 tons of P_2O_5. A horizontal cross flow scrubber prevents the fluorine from escaping to the stack.

The silica used to react with the fluorine is unloaded pneumatically into a 100 ton bin. A metering belt is used to feed the silica to the acid.

The Defluorination plant uses four (4) utilities from the complex central system (in addition to electricity):

a. Pond water, used to scrub the fumes from the defluorination reaction.

b. Steam, used to heat and maintain the acid at reaction temperature. The steam is generated at the sulfuric acid plants.

c. Fresh water from the central system of three deep wells.

d. Natural gas from one of three substations serving the chemical and benefication complex. The gas is used to heat air to keep the exhaust ducts above the dew point. This prevents fumes in the condensation on the ducts wall, which would accelerate build up and pluggage.

Start Up

Since the project was completed in three (3) phases, there were also three (3) start ups as each phase was completed.

The first start up comprised X-Train and two of the storage silos.

At the beginning of April 1981 X-Train produced the first tons of Dical. Since the Defluorination unit had not been built, the acid used was transferred from the super acid plant across the complex. This acid was very pure. The product made then was very high in both phosphorus and calcium.

No major problems were encountered. Some adjustments were made after start up to dry and to screen the product to the desired moisture and granule size distribution. These adjustments also resulted in higher production rates.

There were some problems with the pneumatic conveying system installed to feed the limestone to the plant day bin. Those were solved later on by modifying the equipment.

The Defluorination facility was not started until December of 1981. No problems were encountered except the scrubbing water system. The main problem there was the piping arrangement on both the incoming and outgoing pipes at the point where it tied in with the existing piping system. The problem was solved by making the connection downstream of the existing outgoing pump, and upstream of the existing user at the incoming pipe.

At the start up, acid from other sources were compared with the OXY merchant grade acid. To our pleasure, we found that the OXY acid defluorinated in the least amount of time and used the least amount of silica.

It should be pointed out that it is a time consuming to remove fluorine in the solids. We found that the OXY Hemi process acid is the most economical to defluorinate.

The Product

OXY Feed Division markets two (2) grades of Dical and low fluorine Phosphoric Acid.

Typical analysis are as follows:

	18.5% P Grade	21.0% P Grade	Low Fluorine Acid	
% P	18.9	21.3	% P_2O_5	56.0
% Ca	20.0	16.8	% Solids	1.0
% F	0.14	0.17	% F	0.19
Bulk Density	56.0	56.0		

Compared to the animal feed grade Dicalcium and Monocalcium Phosphate made by other companies, OXY's Dical will soon be the leader in the industry.

A Word Of Advice

For the benefit to those who plan to revive an existing idle plant, take in consideration the work that must be done to comply with the OSHA and other regulations, non-existing at the time of the original plant construction. In our particulate project, a lot of work was allocated to upgrade the safety of the plant to the present regulations, and to our inhouse safety standards.