

A Technical Review of the Improved Hard Process

Presented at
The Fertiliser Association of India
International Technical Conference
April 21-22, 2011
New Delhi, India

Presented by

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1. Abstract

Historically phosphoric acid has been made from phosphate rock by reaction with sulfuric acid (the Wet Process) or a high temperature electric furnace carbon reduction (the Electric Furnace Process). Economic concerns led to the closure of most Electric Furnaces and the Wet Process came to dominate.

In 1960, Lapple demonstrated that a rotary kiln had the ability to replace the energy supplied by electricity in the Electric Furnace Process with energy generated by carbon. This concept was advanced by many researchers but all failed because of melting problems that Dr. Hard later overcame in 1981. The process employs a rotary kiln reactor and was proven in pilot plant testing.

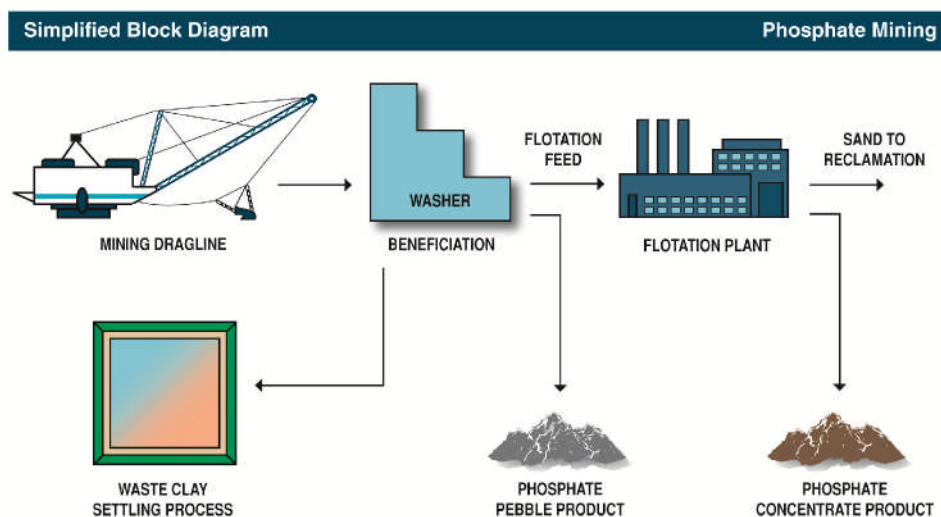
In 2003, Dr. Joseph A. Megy restarted R&D on the Hard Process, and made additional discoveries that led to the Improved Hard Process (IHP), and today design of a semi-commercial demonstration plant is in progress.

The IHP is claimed to be able to process low-grade phosphates and to produce phosphoric acid at lower cost than by the Wet Process. This paper presents the results of a Third-Party review of technical aspects of the process, suitable phosphate reserves, and environmental issues.

2. Technology Overview

The Improved Hard Process (IHP) is an alternative to the most widely used phosphate processing technology, the Wet Acid Process (WAP), and to the less used and more energy intensive Electric Furnace Process (EFP), which due to its high energy costs is currently used in very limited situations to produce high cost, high purity acid.

Both WAP and IHP use phosphate rock which has been mined in either as part of integrated producers' operations, or purchased from a miner of phosphate rock. Following is a conceptual diagram of a typical phosphate mining operation.

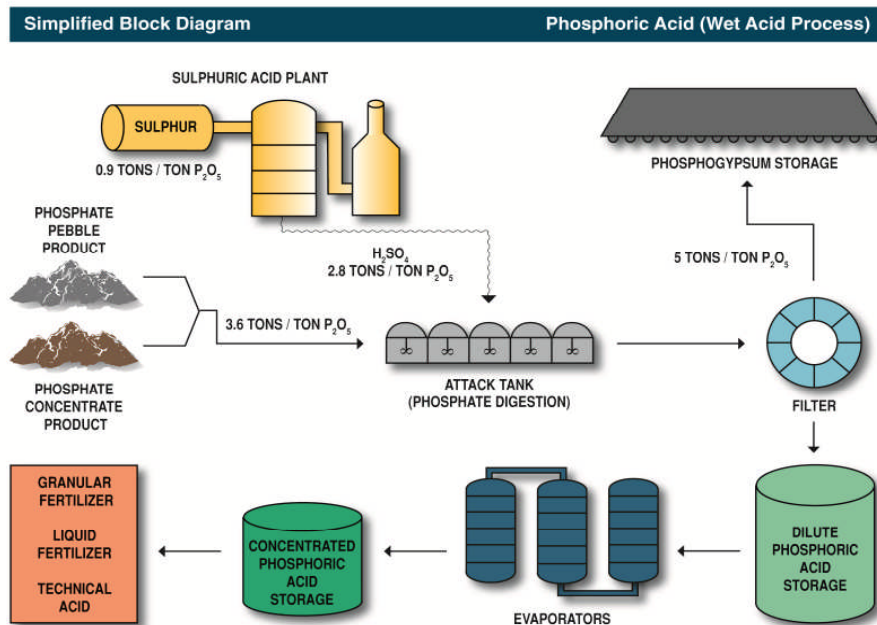


Phosphate mining consists of stripping overburden from the phosphate bearing ore then beneficiating the ore using simple size separation techniques in a washer or other device and/or more sophisticated flotation methods to extract remaining phosphate values that are economically recoverable.

While these mining processes are common to both WAP and IHP, the quality required in the phosphate rock product (and therefore the extent to which beneficiation steps are necessary) and the minimum specifications of ore fed to this mining process vary markedly between IHP and WAP. IHP's ability to process low quality ore is a key to its competitive advantage as will be discussed in this document.

2.1 Wet Acid Process

Most phosphoric acid is produced by the Wet Acid Process (“WAP”) where phosphate rock is reacted with sulfuric acid to make Filter Acid with nominally 28% P_2O_5 phosphoric acid using the Dihydrate process or 42% P_2O_5 using the Hemihydrate process. The main byproduct is phosphogypsum that is about five times the weight of the phosphoric acid produced. All phosphogypsum waste from WAP plants in the U.S. must be permanently stored and monitored on site in large piles known as phosphogypsum stacks.



The Filter Acid is concentrated by evaporation to 50 to 54 % P_2O_5 and reacted with ammonia to make the dominant solid fertilizer products diammonium phosphate and monoammonium phosphate (“DAP” and “MAP”). The Filter Acid can be further concentrated to 54% and clarified to make merchant grade acid (“MGA”). Another fertilizer product is granular triple super phosphate (“GTSP”) made by reacting phosphate rock with phosphoric acid. MGA can, in the case of purer acid products produced from high quality phosphate rock, be further concentrated to 68-70% P_2O_5 to produce super phosphoric acid (“SPA”). SPA can be reacted with ammonia to make fluid fertilizers. Wet process phosphoric acid can also be further purified using solvent extraction technology to make purer acid including technical-grade acid (“TGA”) and food-grade acid (“FGA”).

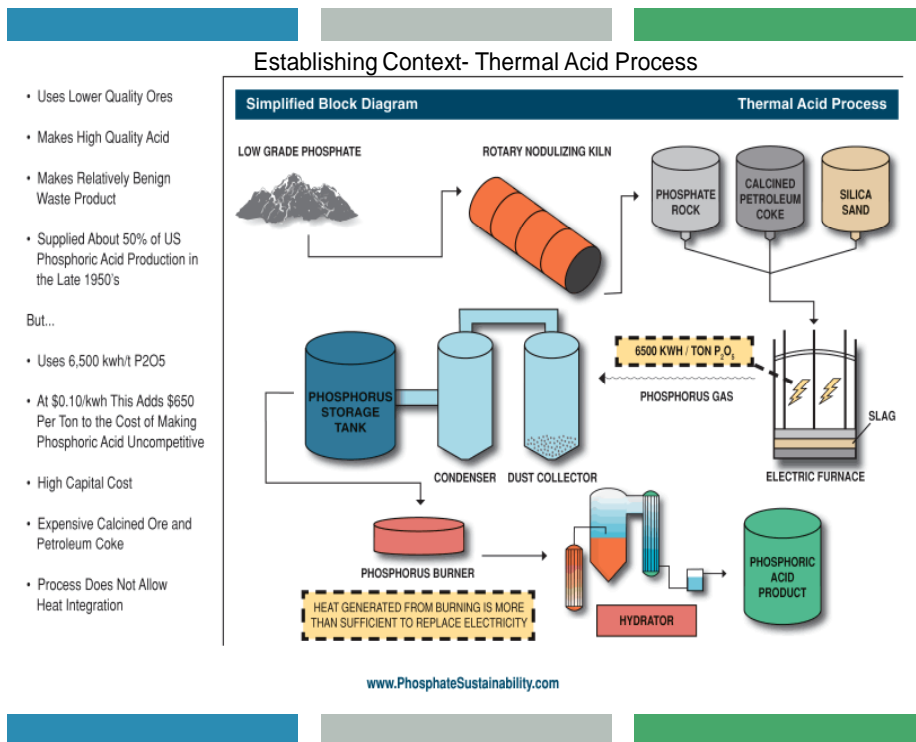
Except for a period in the mid twentieth century when the electric furnace process produced nearly as much phosphoric acid as WAP plants, WAP has been the dominant manufacturing method for producing phosphoric acid. WAP, however, requires highly beneficiated ores as a raw material with relatively few impurities, and makes a relatively weak and impure grade of phosphoric acid with serious environmental impacts.

2.2 Thermal Processes

The electric furnace thermal process and the kiln-based phosphoric acid process are both thermal processes that can be used to produce high quality phosphoric acid from relatively low grade phosphate ores. The following describes the history, principle attributes and competitive advantages and disadvantages of each thermal process.

2.2.1 Electric Furnace Process

The Electric Furnace Process, developed around the turn of the twentieth century, uses electric resistance heating to reduce elemental phosphorus from a mixture of relatively low grade, nodulized phosphate rock, silica, and calcined petroleum coke as the reducing agent. The reaction takes place at about 1600°C, liberating the phosphorus as metal in the gas phase. The reduction reaction is highly endothermic and heat to sustain the reaction is supplied by electrical resistance in a three phase submerged graphite electrode furnace. The phosphorus vapor is condensed to liquid phosphorus metal and then oxidized in a phosphorus furnace to form P₄O₁₀ gas. A hydrator converts the gas to a highly pure and concentrated phosphoric acid.



The phosphoric acid produced using the Electric Furnace Process is of high purity and the waste byproducts are mainly inert slag. The Electric Furnace Process, however, achieves poor thermal efficiency because of the lack of heat integration between the phosphate reduction reaction, which requires a net 6,500 KWH of electricity to make a ton of phosphoric acid (P₂O₅), and the phosphorus oxidation reaction (to produce P₄O₁₀ gas) which is highly exothermic. Rising electrical energy costs in the 1970s resulted in the decline of acid production using the Electric Furnace Process for all but the highest value applications.

2.2.2 Initial Kiln-Based Processes

In the 1950's Dr. W.C. Lapple determined that the rotary kiln was a process vessel with characteristics that could achieve heat integration between a highly endothermic reduction reaction and a highly exothermic oxidation reaction occurring in the same vessel. Unlike the Furnace Acid Process, practically all of the heat required for a kiln phosphoric acid process is supplied by the oxidation of products from the reduction reaction. Lapple reasoned that use of the same raw materials as were consumed in the furnace acid process in a rotary kiln process vessel would accomplish the goal of heat integration. For reasons that were not understood at that time, however, the kiln's charge melted and resulted in process failure.

2.2.3 The Hard Process

In the late 1970s and early 1980s, Dr. Hard and Dr. Megy of Occidental Research Corporation made important discoveries with respect to the kiln-based process. The most critical discovery that occurred was Dr. Hard's insight with respect to the importance of excess silica in feedstock formulations and the role it plays in inhibiting the melting of kiln solids. Dr. Hard's solution to the melting problem was to use much higher silica levels in the feed formulation. This process was patented and became known as the "Hard Process."

Although pilot plant tests validated that the Hard Process produced high quality phosphoric acid the Hard Process had lower thermal efficiencies and throughput and higher costs than desirable, offering only a limited margin improvement over WAP.

2.2.4 The Improved Hard Process

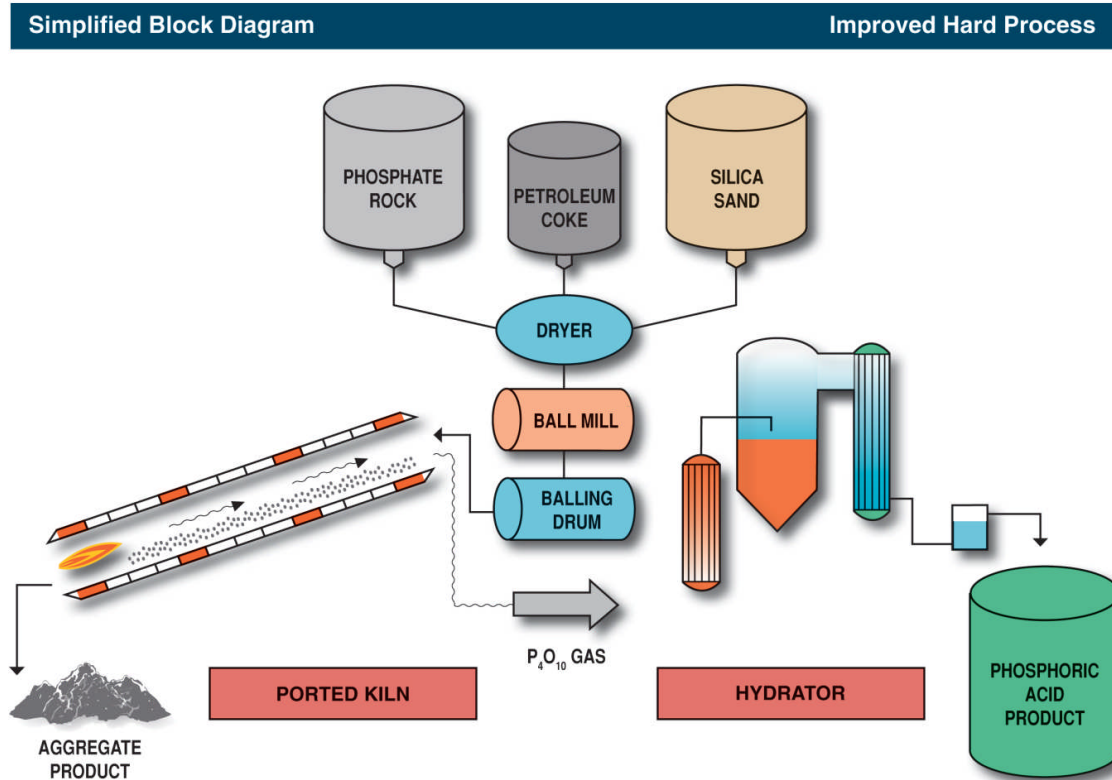
In 2002, Dr. Megy formed a research company called JDC, Inc and reactivated a research program seeking improvements to the Hard Process. A number of significant discoveries were made that quintupled the throughput capacity, dramatically improving the economic and operational profile of the technology.

JDC makes the following claims for the Improved Hard Process compared with the Hard Process:

1. A 30% increase in phosphate in the feed formulation, decreasing unit grinding costs and increasing kiln product throughput.
2. A reduction of kiln residence time to ninety minutes. When combined with the increase in the feed formation set forth above, the reduction in kiln residence produced a five-fold increase in throughput as compared to the Hard Process.
3. A lower and wider operating temperature range of kiln operation, from a minimum of 1180 °C to an effective operating range of 1280-1340 °C, which is less demanding than the higher temperature used in the pilot operation of the Hard Process. The use of a ported rotary kiln allows better control of the temperature profile and extends the reduction zone over a much greater fraction of the kiln.
4. Use of green petroleum coke in place of more expensive calcined petroleum coke.
5. Controlled oxidizing off-gas that allows the process to be run without an afterburner, reducing supplemental fuel requirements.
6. An improvement in process phosphorus yield to over 95%.

3. IHP Process Description

Low-grade phosphate rock, petroleum coke, and silica sand, are fed at accurately controlled flows to a Dryer to remove moisture. The solids leaving the dryer are screened to remove large lumps and the material passing through the screen is finely ground in a Ball Mill.



The ground material is formed into balls in a Balling Drum. Fines and oversize are returned to the Balling Drum and product sized balls are fed to the Ported Kiln.

Air is heated by burning natural gas and blown counter-currently to the flow of material in the kiln. The P₄O₁₀ gas is liberated and passes to the Hydrator where it is absorbed in water to form phosphoric acid. Gases leaving the Hydrator are further scrubbed in a Venturi Scrubber, Cyclone/Scrubber and mist eliminator to recover product not absorbed in the Hydrator. Vent gases are then scrubbed with lime slurry, and then pass to a Flue Gas Desulfurization unit for final cleaning.

Product phosphoric acid is cooled, filtered to remove any suspended solids, and stored ready for shipping.

4. Third Party Technical Review

KEMWorks, recognized as a leading consulting and engineering company in the phosphate and fertilizer processing industries was commissioned by JDC to make a third party technical review of IHP technology. No capital or operating costing was part of this review. KEMWorks assembled a review team that included industry experts from the Florida Industrial Phosphate Research Institute (FIPR Institute) as well as former employees/consultants to Jacobs Engineering, the World Bank and Metso. This third party study is a technical review of IHP, including the development of the process and prospects for success, key potential users of the technology and detailed thermodynamic calculations intended to characterize risks associated with kiln operation.

4.1 Phosphate Resources

The availability and suitability of phosphate resources was reviewed by Jerry Cape. Jerry has a BSc in Mining Engineering and has been an independent consultant since 1974 providing phosphate development services from prospect into production. This work has included project appraisals for

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financing, acquisition, joint ventures, bankruptcies, privatization, and expert witness assignments. He has seasoned expertise in reserve evaluations, mine planning, feasibility studies, financial analysis, project scheduling, estimating, procurement, detailed engineering, cost control, construction, and startup for engineering and owner-operator companies. His total client list is in excess of 50 companies, individuals, and institutions and includes the United Nations, The World Bank, Swiss Bank Corporation, most U.S. phosphate producers, plus many foreign phosphate producers and/or potential producers.

The specification for IHP kiln feed is shown below. If the IHP kiln feed is made from phosphate ore concentrate then the impurities in the concentrate can be about twice as high assuming low impurities in the sand used to bring the kiln feed formulation to specification.

P ₂ O ₅	~ 15%
SiO ₂	~ 50%
Al ₂ O ₃	< 2%
MgO	<2%
Fe ₂ O ₃	< 1.5% *

Three types of deposit were studied:

- Florida feed deltas
- Reject Waste piles
- Existing or new deposits

Other aspects considered in the review were the quantity of the reserves, transport logistics, and availability of petroleum coke.

Reject wastes of past or current phosphate mining operations are a favorable target for the IHP as well as phosphate deposits with high silica, organics, iron and magnesium impurities.

The USGS projects a sharp decline in US and in particular Florida mine production during the next twenty years as ore suitable for the Wet Process is mined out. The IHP can use ore from the Lower Zone and clay settling areas feed deltas that contain P₂O₅ values. In addition because the IHP can accept a lower grade feed beneficiation losses could be reduced. Using IHP in conjunction with WAP could triple Florida reserves.

	Million tons	
	WAP	IHP + WAP
Current W/R Reserves	600	600
Lower Zone		1,000
CSA Feed Deltas		120
Reduced Float Losses		90
Clay & Tailings Additions		30
Total	600	1,840

Other resources deemed to have potential as IHP feed are:

Reject Waste Piles

- Canada
- Senegal

Existing or New Deposits

- Australia

- China
- India
- Jordan
- Namibia
- Morocco
- Uganda

Several phosphates have been recently tested at Metso's research laboratory in batch tests and proved to be suitable feeds for IHP.

4.2 Front End Operations

Front End Operations were reviewed by Bob Faulkner. Mr. Faulkner has a M.S. Metallurgical Engineering, 1961, Colorado School of Mines and has 48 years of experience in the metallurgical industry. He has extensive experience in plant operations background in management; plant start-up; and evaluation of plant operations. Until 2009 he was Manager of Pyrolysis Testing Process Evaluation, Metso Minerals, and responsible process development and evaluation for all projects related to mineral and waste processing. He has directed start-up of Iron Ore plants, developed tire pyrolysis system, directed process development projects for phosphate rock and steel mill waste.

Front End operations include:

- Raw material preparation (high, low phosphate, coke and clay)
- Raw material drying
- Ball mill grinding, day tank storage
- Mixing
- Balling drum operation
- Band dryer
- Ported Kiln
- Product cooler

Mr. Faulkner concluded that there is nothing in the plant that is not standard operating procedure and equipment. The flexibility built into the plant, especially at the feed prep area ensures high availability of feed to the rest of the operation. The pelletizing and drying operation are standard operations and should provide no operational problems. Small start up upsets will certainly be present but they should only require minimum time to resolve. The kiln operation relies on Metso ported kiln experience, the test work and the computer model. This is not unlike first time plants (new processes) that Metso has started up before. The Elkem plant in Norway was a first time commercial process utilizing ported kiln technology. The Wisconsin Power & Electric, Light Weight plant in Oak Creek, WI a first time process started up utilizing ported kiln technology, and this is just the recent history. Both of these plants went from pilot plant testing to commercial units under warranty. There will be some burps as always with a new plant, new operating crew, etc. but there appears to be no fatal flaw in design or operability of the plant.

4.3 Hydrator

The hydrator is a key item of equipment in the process and has an effect on quality of phosphoric acid produced. Hydrator operation was reviewed by Mr. Mike Lloyd. Mr. Lloyd is Director of Research Programs / Research Director - Chemical Processing at the Florida Industrial and Phosphate Research Institute (FIPR Institute). Until very recently this was the Florida Institute of Phosphate Research (FIPR). He oversees and coordinates the entire FIPR Institute Research Program. Mike manages research projects in Chemical Processing and Phosphogypsum research areas, evaluates research proposals, advises contractors, and guides the direction of research. He is the Principal Investigator on several

projects. Mike advises researchers and industry worldwide in phosphate fertilizer production technology and issues, and has coordinated international conferences. Mike received a B. Ch. Eng., at Clemson University in 1950.

The hydrator system proposed for this process and demonstrated on a continuous, countercurrent kiln pilot plant is based on the processing scheme that has been used worldwide on all phosphorus burning plants since the 1960s. One of the major problems to be addressed when attempting to dissolve P_2O_5 in water is how to wet all the P_2O_5 and this has been accomplished using a high pressure drop venturi scrubber. These systems have long been known as having the capability to collect all of the P_2O_5 in the gas stream to give an exhaust gas that does not contain P_2O_5 . The cyclone/scrubber and the mist eliminator are standard for this type operation.

There was very little dust carryover in the pilot plant work. This is a very positive feature for this processing system since the acid product would meet both chemically pure (cp) and food grade specifications.

The plant will be able to produce merchant grade 75% acid (54% P_2O_5) and there is nothing to indicate that 91% acid (72% P_2O_5) could not be made.

4.4 Kiln

Mr. Zimmer has over forty years of experience in the design of mineral processing equipment. Areas of expertise include combustion and heat and mass transfer as applied to incineration, calcining, drying and cooling. Applications include lime, petroleum coke and iron ore pelletizing. From 1997 till present Bill was Manager of Process Engineering at Metso Minerals Industries, Inc. where he is responsible for process design of equipment and development of computer simulations of rotary and vertical kilns, pre-heaters, and coolers. Mr. Zimmer has a B.S.M.E. from Swarthmore College.

The kiln is fed balls containing phosphate rock, silica and petroleum coke. P_4 and CO are produced in an endothermic reaction under reducing conditions inside the kiln bed. Oxygen present in the freeboard oxidizes P_4 and CO to P_4O_{10} and CO_2 . This reaction is highly exothermic and supplies energy to the endothermic reduction reaction.

Yield limitations in the original Hard process were primarily due to carbon burnout (CBO). Carbon burnout occurs the petroleum coke is oxidized by kiln gases. In batch tests carbon burnout occurred during the heating phase before the balls reached reaction temperature and the reducing reaction started. Carbon burnout was much lower in the larger continuous pilot than the batch pilot plant.

CBO will be reduced by using a ported-kiln. Using a ported-kiln will allow the heating period to be shortened and reduce carbon burnout.

Scale-up will also reduce carbon burnout in several ways. First the surface to volume ratio of the kiln increases to the first power of the kiln diameter. If burnout rate stays the same (i.e. mass of carbon burned out per unit time per area of the bed) the fraction of carbon burned out of the bed is reduced in direct proportion to the increase in kiln diameter. If the kiln length gets longer as the diameter increases, then the velocity of gas in the kiln increases as more solids are being heated. The increase in gas velocity will cause greater CBO. The pilot plant kiln was not efficient partly because the gas velocity was relatively high. Higher burner rates were required to compensate for the relatively higher shell heat losses of a small kiln. Adding these effects together, the larger diameter kiln may have lower CBO but might be partially offset by higher gas velocities.

The blanket that forms on the bed from organic volatiles, phosphorus metal vapor, and carbon monoxide blowing out of the bed also has a favorable effect on CBO, which is increased by the diameter scale up of the kiln. Scale up of the kiln has several independent CBO reducing mechanisms that multiply the resulting reduction of carbon burnout.

The temperature window of the Improved Hard Process is sufficiently wide for kiln processing of the phosphate feed. So long as the agglomerates are properly prepared high yields can be obtained at sufficiently low temperatures to allow high production rates. The throughput rate is constrained by heat transfer. The heat transfer available in a rotary kiln allows high throughputs, but the actual rate achievable under commercial practice will need to be demonstrated in a pilot plant.

The ideal temperature profile was not achieved in the batch kiln tests. The temperature rises and then fall along the kiln length. A ported-kiln will allow the kiln temperature at the desired temperature throughout the length of the kiln. The ported-kiln should allow throughput by a factor of three or more compared to the pilot plant.

JDC's heat and material balance model was compared with Metso' simulator and found to give essentially the same results.

4.5 Co-Product Uses

Co-product uses and properties were reviewed by Mr. Sean Masters. Mr. Masters is District 1 & 7 Concrete Engineer for the Florida Department of Transportation. He is responsible for the quality of the concrete for FDOT in the 17 counties of southwest Florida. Mr. Masters has been with the Department for 15 years. Prior to this, he served 8 years in the United States Air Force as an Imagery Analyst in both Pacific and European Commands. Sean graduated from the University of South Florida in 2002 with a B.S in Civil Engineering and is a licensed Professional Engineer with the state of Florida since 2005. He is experienced in both structural and transportation design. He is currently a member of a National Cooperative Highway Research Program committee researching improved simulation models for predicting bicycle and pedestrian demand.

The Improved Hard Process (IHP) makes a silica co-product aggregate with several anticipated uses. The heat from the IHP fuses the material remaining in the kiln feed nodules into porous balls of uniform size. The actual chemical makeup and physical properties can vary due to the original material used with the IHP.

The two main uses for IHP aggregate are:

- for use in concrete
- as a soil amendment.

The IHP aggregate for use in concrete demonstrated many positive factors: it is light weight; is uniformly spherical in shape which should result in a higher compressive strength; uncoated IHP aggregate appears to have a good bond to the concrete paste. IHP aggregate will be tested with larger quantities produced in the demonstration plant.

IHP aggregate has many features that lend itself to its use as a general soil additive. It is chemically inert so it does not leach toxins or degrades over time. It is light weight so it is easy to transport and small and round so it is easy to spread. Finally, its porous structure will absorb water from saturated soils while releasing the moisture as it dries out.

The IHP aggregate was able to transform an extremely poor strength soil into a usable material with a three hundred percent strength gain. It should also be noted that the values generated do not show

the maximum strength gain due to the varying moisture content. The final LBR value and corresponding density would certainly be higher due to optimizing the moisture in the soil. There room for further research in this area.

4.6 Co-Product Radiological Aspects

Co-product radiological aspects were reviewed by Dr. Birky who is currently Program Director at the Florida Industrial and Phosphate Research Institute, a Faculty member at the University of South Florida Polytechnic, and Senior Health Physicist at Applied Environmental Consulting, Inc. He received a Ph.D. in Health Physics in 1997 from the University of Florida, Gainesville, FL. Brian made several relevant presentations at the 6th International Symposium on Naturally Occurring Radioactive Material (NORM VI), Marrakech, Morocco, March 22-26, 2010, including “Phosphogypsum use in road construction” and “Safety, radiation protection and management of radioactive wastes in the phosphate industry, the three layer conceptual model”. Brian is a Plenary Member of the Health Physics Society and Past President of the Florida Chapter of the Health Physics Society.

The assessment was based a review of a report by SENES. SENES emphasized that this assessment applies to the co-product generated in the pilot-plant stage of development, and that it should not be considered for regulatory approval of the uses. This is a prudent approach at the current state of development and considering the fact that only a limited radiological data set is available for analysis.

The two uses considered in the assessment are:

- Soil stabilization (e.g. highway base); and
- Concrete-based applications (porous concrete and direct drainage uses).

SENES states that due to the very low radon emanation characteristics of the co-product, potential exposures to radon and decay products would not result in incremental radiation doses above background.

FIPR Institute Evaluation: We agree with the summary conclusion based on the data provided, but recommend more extensive radon emanation testing when more co-product is generated in the development phase. The evidence base should include more data points from different production batches with data verification from independent laboratories.

SENES states that potential exposures via inhalation or accidental ingestion of the co-product would result in insignificant exposures.

FIPR Institute Evaluation: We agree with the summary conclusion for the co-product as generated, but reserve judgment for applications which may use processes that alter the material, such as fine grinding. However, it is not expected that such processes will be required in the proposed applications.

SENES stated that exposure to external gamma radiation would be the only potential exposure pathway of significance. A radiation dose of 11 mrem/y was estimated for workers who would be directly exposed to the undiluted co-product during application. Exposures under different exposure scenarios would likely result in lower doses. The estimated dose of 11 mrem/y estimated in this analysis is a small fraction (about 3%) of the average U.S. natural background radiation dose of 310 mrem/y and about 10% of the dose limit of 100 mrem/y recommended for the maximum exposed members of the public from sources of naturally occurring radioactive materials.

FIPR Institute Evaluation: Exposure to external gamma radiation is likely to be the pathway of greatest significance. Even under extremely conservative assumptions, the dose is less than the public dose limit and it is more probable that the dose will be only a small fraction of it.

SENES conclusion from the cover letter: “The proposed uses [of the co-product aggregate] would not result in any significant radiological exposure either to workers or members of the public.”

FIPR Institute Evaluation: We agree with the summary conclusion based on the pilot plant data provided, and the limited end uses evaluated.

4.7 Product Uses

Mike Lloyd also gave an opinion on product uses for IHP phosphoric acid. IHP acid will be very pure and has a low minor element ratio (MER). MER is the ratio of aluminum, iron, and magnesium to P_2O_5 . Acid as pure as this could not be used to manufacture commercial DAP (18-46-0) as impurities are needed to promote granulation. However, it would be possible to crystalline DAP with the analysis of 21 - 53.4 - 0.

The most practical solid fertilizer use for this acid would be blended with normal Merchant Grade Acid made in the wet process that has relatively high impurities (high MER). The blend would an acceptable MER (neither too high to meet grade or too low to granulate) that could be used to manufacture DAP. In all likelihood if this acid were used in conjunction with wet process acid, it would be possible to use a more impure rock feed for the wet process acid plant. The higher strength acids (above 75%) could be used for liquid fertilizers with a high polyphosphate content that will allow the manufacture of the highest possible analysis liquid and fluid fertilizers. Acid of this purity could easily be used for food grade applications.

5. Conclusions

There are many suitable phosphate deposits with potential feed for the Improved Hard Process and several of these have been recently tested in batch mode at Metso. Suitable sources include reject waste piles, Florida clay settling area feed deltas, and low-grade deposits with high silica low aluminum content.

In the front-end there is nothing in the plant that is not standard operating procedure and equipment. The kiln operation relies on Metso ported kiln experience, the test work and the computer model. There appears to be no fatal flaw in design or operability of the plant.

The hydrator system proposed for this process and demonstrated on a bench-scale pilot plant is based on the processing scheme that has been used worldwide on all phosphorus burning plants since the 1960s.

The IHP aggregate for use in concrete demonstrated many positive factors: it is light weight; is uniformly spherical in shape and uncoated IHP aggregate appears to have a good bond to the concrete paste. IHP aggregate also has many features that lend itself to its use as a general soil additive.

A Review of SENES Report agreed with its conclusions that there would be very low radon emanation characteristics of the co-product. The proposed uses of the aggregate would not result in any significant radiological exposure either to workers or members of the public.

IHP product acid can be made from low-grade phosphate it could be used to sweeten MGA and allow the wet process phosphoric acid to use a lower grade of rock thus extend the life of reserve and allow simpler beneficiation with lower losses. The higher strength acids (above 75%) could be used for liquid fertilizers with a high polyphosphate content that will allow the manufacture of the highest possible analysis liquid and fluid fertilizers. Acid of this purity could easily be used for food grade applications.

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