IJC'S EXPERIENCE WITH HEMIHYDRATE PROCESS

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Abstract

Indo Jordan Chemicals Company (IJC) is a joint venture company promoted by M/s Southern Petrochemical Industries Corporation Ltd (SPIC), India, M/s Jordan Phosphate Mines Company Ltd (JPMC), Jordan and The Arab Investment Company (TAIC), Saudi Arabia. The Industrial Complex has been set up in Eshidiya, Jordan adjacent to the Phosphate mines of JPMC.

IJC operates a Phosphoric acid plant, based on Hydro Agri's Single stage Hemi hydrate process with a name plate capacity of 2,24,000 MT per annum, a Sulphuric acid plant with Monsanto's DCDA process with a name plate capacity of 6,60,000 MT per annum and associated utilities and Off-site facilities. Situated next to the mines, it handles rock of various grades and varying level of impurities.

The di-hydrate process served as the standard for phosphoric acid industry for many decades. It is however less energy efficient as the rock feed has to be finely ground and the acid produced is dilute at 28%P2O5 which needs more energy to be concentrated.

Hemi process produces acid at high concentration (42 % P_2O_5) of phosphoric acid, which enables easier conversion in to downstream fertilizers. The gypsum crystals exist in the hemihydrate form ($CaSO_4.1/2H_2O$) consuming lesser amount of water. The process is suitable for processing impure rocks with 60% Tri calcium phosphate (TCP).The process is energy efficient as there is no fine grinding of the raw material used .Acid of higher strength of 42% P2O5 resultant from the process, reduces the energy spent on concentrating the acid to the final merchant grade concentration of 54 % P_2O_5.

The acid is more pure with lesser amount of impurities like Sulphates, Aluminium, Fluoride and solids and consequently lesser sludge formation .This reduces the task of managing the tanks, whose cleaning can be cumbersome.

However it's all, not a bed of roses, there are some thorns too. The hotter conditions in the Reactor aggravate the corrosion of agitators and filter metal surfaces. The belt filter rubber is also one casualty affected by the higher temperature, aggravated by the defoamer.

This paper discusses the

- Processes, comparison and advantage of the hemihydrate process
- Operation with low grade Rock phosphate
- Utilization of Fixed laterals instead of curbs in the Belt filters

Processes for manufacture of phosphoric acid:

The various processes for producing phosphoric acid are named after the type of gypsum produced. There are basically three types of processes which are industrially important. They are the Di hydrate process, Hemi hydrate process and the Hemi- Di process .In di hydrate form the gypsum crystals are formed at lower concentration and temperature while in hemi hydrate process the gypsum crystals are formed at higher concentration and temperature. The di-hydrate crystals have two molecules of water per calcium sulphate molecule while the hemi-hydrate crystals have half a water molecule per calcium sulphate molecule. Operating conditions in the di hydrate process stay just below the hemi-di boundary while in the hemi-hydrate process it stays well above the boundary in the stable zone in the Nordengren diagram. In the Hemi-Di process, there is an additional transformation tank and filter added to the hemi hydrate process. The gypsum sent out of the plant is the Di hydrate crystals.

Dihydrate process :

Dihydrate produces acid at 28% P_2O_5 so that further concentration to 54% P_2O_5 is needed for producing fertilizers.

The advantages are :

Flexible operation and long experience

Efficiency of about 95 %.

Wet Rock grinding possible

Lower grade of Stainless steel enough for handling vessels.

Disadvantages are :

Needs fine grinding, as all particles should be less than 0.42mm

Larger acid concentration units for producing merchant grade

Acid has more Aluminum and Fluorides as impurities.

More steam and cooling water requirements.

Post precipitation before and after evaporation.

Hemi hydrate process :

Hemi hydrate process produces acid at 40 to 42 % P_2O_5 directly.

The advantages are :

Rock grinding (as all particles are coarser and less than 1mm size is adequate) and evaporation requirements are minimum.

Capital cost is lower than di plants.

Lesser steam and cooling water requirement.

More pure acid with lower sulphates, Aluminum and Fluorides.

Limited post precipitation

Dis Advantages are :

Lesser Efficiency at 90 – 92 %

Higher temperature operation causes more corrosion.

Scaling in the filter zones where hemi to di transition takes place.

Tighter water balance

Hemi - Di process:

This process produces acid at 42 % - 45 % P_2O_5.

The advantages are :

Higher efficiency (98 % and above)

Gypsum more pure.

Recrystallisation tank acts as buffer tank for startups / shutdowns and mechanical losses from the HH Filter.

All the other advantages of the hemi hydrate process.

The disadvantage are :

More equipments are added – two stage filtration.

and power consumption more.

Capital cost being higher than either hemi or di process.

Process Comparison :

Parameter	Di hydrate	Hemi hydrate	Hemi- Di
Capital cost	Plant involves grinding, evaporator and bigger cooling tower and a clarifier added to a hemi plant. Reactor $2m^3$/t P_2O_5	20% lower than di plant; Reactor is smaller and plant can be loaded to have a volume of $1.5m^3$/ton P_2O_5	Same as di plant due to added reactor and filter
Rock grinding	50kw/ton P2O5	No grinding power	No grinding power
Evaporation	Bigger units as 2tons water is to be evap per ton P2o5 for conc from 26% to 54%P2O5	Smaller unit as 0.5tons water is to be evap per ton P2o5 for concn from 42 to 54 % P_2O_5	Smaller unit ~ 0.45 Ton of water/Ton of P_2O_5 for concn from 43 to 54 % P P_2O_5.
Cooling water	More qty needed- cooling water at 33'c	Less and cooling water can be higher at 38'c	Same as hemi plant.
Operating cost	More due to grinding (50KW) and evaporation units (300KW) equivalent power	Less due to smaller evaporator and steam needs.	Higher due to 2 reactors and filters - Power 30KW/T p2O5 more than Hemi plant.
Filter recovery	At 95%	Less about~ 92%	Highest at 98%
Product acid strength	28 % P_2O_5	40 ~ 42 % P_2O_5	42 % ~ 43 % P_2O_5
Purity suitable for DAP	Impurities more can affect high grade fertilizer production	Purer acid suitable for direct use in DAP manufacture.	Same as hemi
Water consumption	More due to di-hydrate cake and filter product is lower conc of 28%.Gypsum storage needs liner	Hemi hydrate has lesser water and gypsum is self drying and acid of 42%p2o5 has less water. Gypsum storage is easy	Same as hemi

Hemi hydrate process :

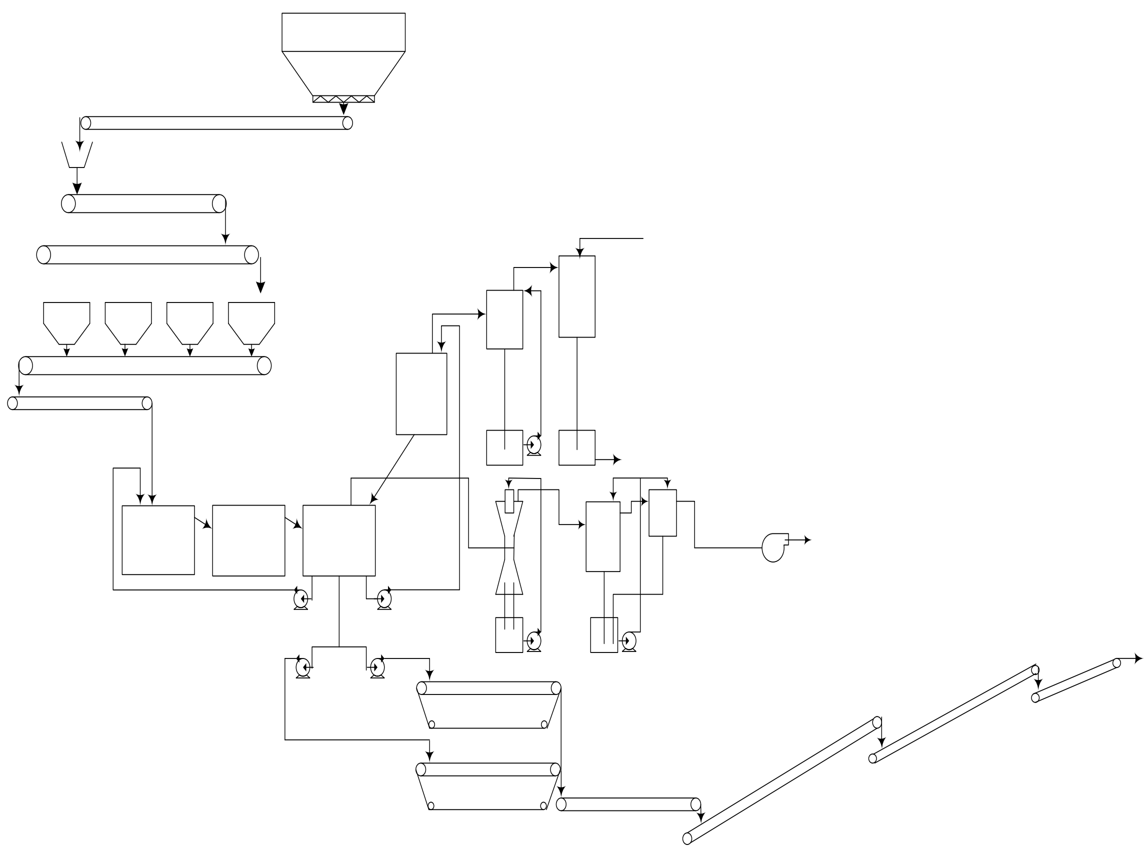
General process description:

The hemi hydrate reaction system consists of three cylindrical tanks of equal volume and dimensions. The first two Reactors are named R1A and R1B.The third one is R2.

Reactor R1A and R1B are deficit in sulphate and have been designed as two separate tanks to enhance the release of gas produced by the reaction of rock and acid thereby improving process control. Phosphate rock is discharged into Reactor R1A, while 98.5% sulphuric acid is fed to Reactor R2 through the concentric static Acid Mixer where it is mixed with the return acid flows (approximately 35-38% P2O5 phosphoric acid from filters).

Reaction slurry overflows from Reactor R1A to Reactor R1B then to Reactor R2 through launders.

From Reactor R2, Part of the slurry is recycled to Reactor R1A by a Recirculation Pump, so that only 40% of the total CaO (w/w %) fed to Reactor R1A is precipitated. Further from R2, part of the slurry is cooled in a Flash Cooler by evaporation of water from the slurry under vacuum to 94°C. This flows back through a down leg sealed in R2 and helps in maintaining the temperature of Slurry below 100 degrees Celsius. The free sulphate level in Reactor R2 in its liquid phase is controlled at around 2.0 w/w % Slurry from Reactor R2 is fed to Hemihydrate Vacuum Filters.



Operation with low grade Rock phosphate :

There are 3 types of Rock supplied by the JPMC mines plant. They being

Schedule A - Commercial Rock 73 - 75 BPL Dry Phosphate Rock

Schedule B - Commercial Rock 70 - 72 BPL Wet phosphate rock having 14 - 18% moisture

Schedule D - Sub commercial 60 - 65 BPL Wet Phosphate Rock containing 5 ~10 % moisture.

IJC plant is designed to handle predominantly A or B with a plant efficiency of 94%. The plant is operated with Schedule D rock since November 1998 as a blend with D content up to 70% of the Total Rock used. D rock has a lower P_2O_5 of 27.5% as against the B rock which has a p2o5 of 32.5%.However it is found that the plant operation is more stable only when the D Rock was blended and used along with the commercial Rocks and not separately.

Increased erosion of the rotating equipments and pipelines and lower filtration rates were experienced when D rock alone was processed. The blend of sub commercial Rock along with the Commercial Rock was performing equivalent to the commercial Rock at proper operating conditions except for the lower plant efficiency and higher Rock consumption.

The design specification of Iron and Aluminium are 0.5% and 0.6% While the average figures in the D Rock are 0.45 % and 0.76 %.when the Aluminium content is less than 0.6% in the Rock fed to the Reactor then the filtration rate gets lowered. To counter this the manual addition of Kaolin containing about 20 % Alumina is being resorted to and hence this 0.6% seems to be the lower limit for the hemi hydrate process.

The Rock D is blended with Rock A or B and used as it improves the filtration rate .IJC has a total filtration area of 160m2 from the two belt filters and production capacity of 4.5 TPD/m^2 is achieved ,equivalent to 720 TPD.

Operating experiences with lower grade rock phosphate:

The Sub commercial Rock used has about 80% of the feed less than 1mm size while the plant is designed for 80% less than 0.5mm size. The coarse material above 1.5 mm size does not react well and some of the coarse unreacted /partly reacted particles get accumulated at the bottom of the First Reactor. This necessitates Reactor draining once in a day for about 10 minutes as otherwise the Carbon brick lining inside the Reactor gets eroded fast. The drained liquid is strained through coarse strainers to a sump from which the filtrate is recovered back to the Reactors. To reduce this draining and to avoid the coarse rocks which will not react fully, attention is being given to maintain the size distribution of the Rock fed to the Reactor.

The solid content in the Reactor slurry was increased by 1% to handle the additional quantity of solids generated on account of processing lower grade Rock. Marginal increase in scaling inside the flash cooler and slurry inlet and outlet ducts was noticed. There was a slight increase in the whitish silica deposits in the scrubbers and vapor ducts increasing the frequency of cleaning as the sub commercial Rock has about 10 to 15% silica. The quantity of gypsum handled also increases from 5 tons to 6 tons per ton of P2o5 due to the higher impurities present in the Rock.

Utilization of Fixed laterals instead of curbs in Belt Filters :

IJC uses two belt filters which were fixed with curbs on the belt to avoid slurry spillage. The hemi hydrate process at IJC employs Rock of varied composition as it is situated next the mines. The organic matter and CO_2 are responsible for the foaming and defoamer of about 10kg /ton of P_2O_5 is used .These defoamers potentially harm the rubber .Failure of the curbs on the belts were experienced in the Year 2007 and steps to cold vulcanize the curbs on to the used filter belts were not successful. Slurry spillage from the belts from the damaged curbs was hampering the production .Hence the concept of fixed laterals was thought of and the supplier EIMCO of ITALY was contacted.

Laterals were installed in Filter 2 in October 2007 and subsequently in Filter -1 in Feb 2008 .The fixed laterals as the name implies, are fixed to the frame of the belt and do not move .The belt moves underneath the fixed laterals made of Stainless steel while the filter cloth slides along the sides of fixed laterals .To avoid the slurry spillage through the gap between the laterals and the belt ,two more layers have been provided over the metal laterals in the form of a rubber sheet over the metal and a Teflon sheet over this rubber .The cloth used to run sliding over the Teflon sheet . To take care of the hot slurry and expansion of the Teflon, gaps had been provided in between the Teflon sheets. Sometimes the cloth enters between the Teflon sheets and gets torn and also caused slurry to spill from the belt filter.

The cloth life reduced to around 15 days instead of the traditional 45 days life. To counter this, an additional rubber layer (used conveyor belt) was put over the Teflon sheet and this has improved the cloth life to around 30 days. Further cloth guiding rollers were fabricated and installed.

During the initial start up heavy air ingress through the both sides of the belt was noticed as the laterals were fixed at the places where the belt grooves had begun. Air was being sucked in through the grooves causing poor filtration .This was corrected by shifting the laterals widthwise towards the edges of the belt so that laterals are installed at the edges of the belts. When the weight of the slurry cake is less due to lower feed rates, there is air ingress through the sides resulting in loss of vacuum and filtration problems. The cloth gets torn off at the edges and the cloth renewal frequency has increased subsequent to installation of the fixed laterals.

Modifications in the cloth by providing suitable reinforcement layers in both the edges of the clothes is planned .Further lowering of the laterals and changing of the angle of the laterals over the belt minimizing the gaps is being planned to improve the vacuum.

<u>Fixed lateral protections supported by a Stainless Steel frame, without curbs</u>

The drainage belt is not provided with the traditional two edge flanges, but it is provided for the entire length with fixed lateral protections in Teflon, supported by a frame in SS material.

Laterals in line

The lower part of the lateral protections, close or in contact with the drainage belt, is in rubber material. It is fixed between the stainless steel frame and the Teflon. Another rubber layer in the form of used conveyor belt has been provided over the Teflon and the cloth slides over this rubber.

Conclusion / Summary:

- The soaring fuel cost has a major impact on process selection. Hemi and Hemi – di are more energy efficient. Further the added benefit of lower energy consumption is that it also combats global warming. The acid produced from the hemi plant at a higher intermediate concentration of 42 % enables easier production of merchant grade phosphoric acid of 54% as compared to a Di plant which produces an intermediate acid of 26 % P_2O_5.
- IJC has been using a substantial portion of sub commercial Rock due to the selection of the Hemi process which is able to tolerate a bigger Particle size, with lower purity of P_2O_5 and with higher amount of silica.
- The provision of fixed laterals instead of curbs has made the belt to operate without fear of the failure of curbs. However the provision of fixed laterals attaches more importance on the maintenance of the filter, as the cloth should always run without sway .The cloth tracking device and all the supporting rollers for the cloth and belt should be in good working condition to ensure that.

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