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Experience with Hemihydrate Process to Produce Premium Quality Phosphoric Acid

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Abstract

Indo Jordan Chemicals Company (IJC), located at Eshidiya; South Jordan neighbouring with Jordan Mines and Mineral Company (JPMC) LLC, was established in the year 1997 and has been performing at its best to date. The Company is producing premium quality phosphoric acid using hemi hydrate (HH) process and exporting to its esteemed customers.

Using low quality rock phosphate, achieving 118 % plant capacity utilization with 98 % on-stream factor to produce and meet the customers' premium quality export is the main focused discussion in this paper. Over a period of time, IJC faced a tough challenge on rock phosphates quality as JPMC has downgraded its rock phosphate supply to its internal customer without compromising quality of commercial grade rock phosphate to international customers even in variations of phosphorites purity in operating mines in Eshidiya.

This paper is intended to share the IJC's experiences on HH phosphoric acid plant operation to handle low grade rock phosphates and achieving best quality of phosphoric acid. Its journey towards continuous improvement on plant's reliability, productivity and efficiency may be useful for other producers worldwide. Other than fertilizer grade acid, IJC has started its journey to attract its Asian and European customers for animal food grade phosphoric acid. With continuous endeavour and bench scale study, quality of food grade acid has been brought well within the norms set by international bodies.

Methodology of handling of sub-commercial rock, up-gradation of MOC of equipment to the latest trend, maintenance practices & performance monitoring of equipment in predictive and proactive manner, revision of standard operating procedures, industrial best practices and training of employees with latest technological advancement are few success stories of the company and have been discussed in the paper.

Key words : Phosphoric acid, hemi hydrate process, rock phosphate, reliability, quality

1. Introduction

The project in Eshidiya complex consists of 224,000 MTPA (as 100 % P_2O_5) phosphoric acid plant, 660,000 MTPA dedicated sulphuric acid plant and associated utilities & offsite facilities and an acid storage facility at Aqaba sea port.

IJC plant is designed by M/s Yara to handle predominantly A & B grade rock phosphate supplied by nearest JPMC mines with plant efficiency of 94%. After commissioning, IJC started production from 1997, but plant was started operating with different grade (schedule D) rock since November 1998 as a blend with other two types.

IJC faced a tough challenge on rock phosphate quality since JPMC diversified rock phosphate quality due to increased demand of commercial grade rock phosphate in international market and degradation of phosphates purity in operating mines.

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IJC's commitment to supply the high-quality merchant-grade phosphoric acid to the international buyers and strongly withstand its vision & mission of not to deviate processed rock phosphate grade affect the final product acid quality of various satisfied customers.

IJC's policies and concepts evolved and adopted based on the experience and expertise of the multidimensional teams' involvement from the project implementation to operate the plant from complicated conditions to user-friendly directions.

This paper covers various difficulties experienced by IJC to operate the plant more than the name plant capacity and troubleshooting carried out to maintain the premium quality acid.

2. Overview of IJC Phosphoric Acid Plant's Equipment Capacity, Capability and Reliability

IJC HH process designed by Yara is considered to be 1st project of its kind and success story of its performance, capacity utilization and quality of acid had change the mind set of many producers on HH process which considered to be tougher than Dihydrate (DH) process. Basic disadvantage of the process is comparative lower efficiency than DH or hemi dihydrate (HDH) process but it has many other advantages.

We would like to highlight eight pillars of silver lined landmarks.

- Overall capacity utilization.: 118% and marching forward
- Continuous reduction in the specific consumption of raw materials and consumable chemicals in phos acid : (e.g. rock phosphate, sulphuric acid, defoamer, anti scaling agent, raw water and power)
- Fulfillment of quality fronts.: 0% customer complain from fertilizer sectors and marching forward to animal food grade acid
- On stream factors:98%
- On stream efficiency:114%
- Safety compliances: Zero loss time accident, awarded and honoured by BSC.
- Environmental aspect and waste management: awarded non-compliance from Government of Jordan
- Wellbeing to stakeholders: Awarded and honoured by BSC (British Safety Council)

3. Operation with Different Grade Rock Phosphate

IJC, as a merchant grade phosphoric acid producer, is increasingly encountering regularly fall in P_2O_5 grade of rock phosphates and simultaneously increase of impurity content from its parent supplier Jordan Phosphates and Mines Company (JPMC).

Being faced these unavoidable circumstances, shifting from existing operational methodology and adopt the new standard operating procedures (SOP) is a challenge due to the increasing global demand across the globe and to make the business economically viable.

In addition to that there are many changes on quality of acid, set by international fertilizer fraternity, by lowering the limit of minor elements present in acid making stiffer competitions among merchant grade acid producers.

However, IJC has taken adequate and timely measure to meet the changing standards.

3.1 Comparative Evaluation of Jordanian Phosphorites: a Basic Raw material for Phosphoric Acid Production

With changing quality of rock phosphates' over the period of time and the requirements of various corrective measures for processing the rock phosphate without affecting the volume & quality of production with optimum efficiency in IJC HH process of phosphoric acid unit are considered.

In year 1997, IJC was getting basically three grades of rock phosphate from JPMC Eshidya mine as given below :

- **Schedule-A**: Commercial grade rock, 73-75 TCP, dry rock carrying only 2.5-3% moisture.
- **Schedule-B**: Commercial rock for selected customers, 70-72 TCP, wet phosphate having 14-18% moisture.
- **Schedule-D**: Sub-commercial, 60-65 TCP, dry rock having 5-10% moistures.

IJC is obliged to receive and process more non-commercial rock phosphate from the year of 1998.

Following are the present status of rock quality received:

- *Schedule A has been shifted to A2 (which is next down grade level of the export quality A1) :*TCP 68-70, 3-4% moisture and sometime A2 is mixed with S_2 grade rock phosphate from different mining source to standardize A2 quality with respect to TCP but doing so it contributes higher degree of R_2O_3 (Al_2O_3 & Fe_2O_3).
- *Grade B has been modified as A1A3:* It's a mixture of beneficiation plant's filter output of A1 grade rock with A3 grade: TCP-63-65, moisture 15-18 %. Commercial is available only on demand with high moisture for selective customers.
- *Grade D: Sub commercial in nature:* Un- beneficiated, 58-61 TCP, dry but rich in unwanted elements of silica, chloride, Al, and Fe.

Individual rock quality and blended feed rock quality is shown in the **Table 1.**

IJC identified ratio 60:20:20 and or 60:30:10 performs better than other mixing composition.

3.2 IJC Methodology for JPMC Supplied Rock Phosphate Quality Control:

- As a daily routine job, a team from IJC comprising of highly experienced supervisor and quality control team work together with JPMC mines area supervisor to segregate out the different strata of phosphates situated at various screen in mines area.
- Composite samples are brought to lab for analysis and on basis of lab report, either samples are qualified or reported back to JPMC for further improvement with the intended mission of not to suffer the plant with respect of volume of

Table 1. Individual rock *vs.* blended feed quality

Eshidiya, Jo- Mines Phosphates					Ratio of RP, (in PCT) A1A3 : D : A2			
Parameters	UOM	A1 A3	D	A2	1:01:01	50-25-25	60-20-20	60-30-10
Moisture	%, w/w	16.49	2.2	5.25	7.98	10.11	11.38	11.08
TCP / Grade	%	66.99	61.94	67.41	65.44	65.83	66.06	65.52
P_2O_5	%, w/w	30.66	28.35	30.85	29.95	30.13	30.24	29.99
CaO	%, w/w	45.05	45.44	49.51	46.66	46.26	46.02	45.61
Silica	%, w/w	10.68	14.07	12.30	12.35	11.93	11.68	11.86
SO_3	%, w/w	0.54	1.10	0.68	0.77	0.72	0.68	0.72
F	%, w/w	2.74	2.46	2.75	2.65	2.67	2.69	2.66
CO_2	%, w/w	3.40	4.86	2.84	3.70	3.63	3.58	3.78
Chloride as Cl	%, w/w	0.02	0.10	0.02	0.05	0.04	0.04	0.04
Al_2O_3	%, w/w	0.63	0.90	1.35	0.96	0.88	0.83	0.78
Fe_2O_3	%, w/w	0.38	0.78	0.95	0.70	0.62	0.57	0.56
MgO	%, w/w	0.21	0.23	0.17	0.20	0.21	0.21	0.21
Na_2O	%, w/w	0.29	0.37	0.28	0.31	0.31	0.30	0.31
K_2O	%, w/w	0.08	0.10	0.12	0.10	0.10	0.09	0.09
Org. Matter,C	%, w/w	0.1150	0.1050	0.0830	0.10	0.10	0.11	0.11
FCR		0.81	0.51	0.69	0.651	0.685	0.707	0.685
MER		0.04	0.07	0.08	0.062	0.057	0.053	0.052
P_2O_5/CaO		0.68	0.62	0.62	0.64	0.65	0.66	0.66
Reactive silica	%, w/w		60.00					

Sieve size	**UOM**	**A1 A3**	**D**	**A2**
+ 6.3 mm	%	Nil	Nil	Nil
- 6.3 + 4 mm	%	Nil	Nil	Nil
- 4 + 2 mm	%	2.72	7.73	7.10
- 2 + 1 mm	%	6.64	12.27	11.90
- 1+ 0.5 mm	%	23.09	15.88	22.30
- 0.5 + 0.212 mm	%	32.58	31.04	32.80
- 0.212 mm	%	34.97	33.08	25.90

Other Minor elements in Eshidiya RP		
Cd	ppm	4-5
As	ppm	14
Pb	ppm	1-2
Cu	ppm	15-19
Zn	ppm	190-200
Ni	ppm	27
CO	ppm	9
Mn	ppm	330
Cr	ppm	50-66
Ti	ppm	60-70
V	ppm	40-70
Hg	ppm	nil

Table 2. Comparision of an ideal rock with IJC processed rock

Rock phosphate index	Effects on process	Ideal values*	IJC blend values
High P_2O_5/CaO	Gives better yield of process	0.71	0.66
Low carbonate	Less foaming and SA consumption	1.7	3.58
Low organic matter	High organic matter in combination with CO_2 increases foam stability index.	0.04	0.11
Right FCR values of rock phosphate	It affects crystal habit, shapes and size.	0.8	0.707
Low non- reactive silica	Silica required as reactive to minimize ill effect for HF but non-reactive silica effects total reaction process by additional unwanted solid generation and highly erosive to rotary equipment.		65% of total silica in rock phosphate
Low alkali metal impurity	Helps less scaling in equipment especially colder zones of vacuum circuits.	-	0.39
Low cationic impurity	It helps to minimize orthodox scaling in combination with other impurity and less influence on downstream derivatives of phosphoric acid.	-	
Sufficient selective cationic impurity	Some cations have a positive effect of crystal habit modification and helps for better filtrations.	-	
Superior reactivity of rock phosphate	Less needs higher reacting volume and time to complete the reaction.		Jordanian rock phosphates are reactive as mentioned by researchers
Softness of rock phosphate respect to grinding requirement	Low power consumption		No grinding requirement for HH process
Low chloride	Invites corrosion	<0.03%	0.04%
Low MER	Helps low viscosity, low scaling, no side effects in fertilizer	Not to exceed 0.1	0.053

*TOGO high grade rock is taken as reference

production and quality of final acid.

Comparision of an ideal rock with blended rock processed by IJC is given in **Table 2.**

4. Short Description of Reaction, Scrubbing, Filter Operation and Concentration Section

There are some special features of HH reaction and filtration circuits which demands special attention for process control, selection of MOC of equipment, fabric for filter cloth and addition of specialty chemicals to prevent foaming in reactor, chocking of lines and vessels from scaling and reductions of heat transfer coefficients in manufacturing the products.

Due to high temperature reaction, reaction vessels and roof are required for better protection than DH plants.

- IJC faced serious deterioration of reactor roof concrete due to torturing effects of evolving fluorinated gases and hot vapour/fumes generated due to acidulation of reactor slurry from acid mixture situated in 3rd reactor (R2). With continuous benchmarking with other industries, a special quality high temperature bearing resin is started applying in dry surface of reactor roof for last couple of years which had given comparative relief from deterioration.
- Regarding MOC selection of reactor agitators, latest developed austenitic stainless steel are used as per recommendations of OEM suppliers. It is found to be performing better than earlier.
- Regarding pumps, those are connected to reactor; to handle slurry were initially found to be suffering higher rate of erosions and damage of internal lining. IJC has developed a well-recognized design house that has tailor made the pumps spare parts which have improved life to more than double.
- Liberation of obnoxious gases through reaction is more in comparison of other process. Without compromising environment and employed health, MOC selection has been done meticulously with

right specification.

- Gas scrubbing sections are made more efficient by ventury scrubbing system and circulating scrubbing water in 3 stage yields a 50-100 mbar negative draft over the reactor. The gases liberated through final stage to atmosphere are well below the environmental norms as specified by pollution board of Jordan.
- As IJC uses sub commercial rock phosphate as a blending with other veriety e.g. A2 & A1A3 – as per sieve analysis shown before, it contains 6-7% +2 mm particles . These particles, if reside in reactor for prolonged time, damages the rotary parts of agitators and pumps. Therefore daily discard of higher size particle through reactor draining nozzles to sump is an invariable practice.
- In IJC, HH filters are running 15 days nonstop. Scheduled online checking procedures are carried out on daily basis by operation and mechanical PM team and cross checked and verified by inspection team.
- Monthly two times washing of filters are taken for 8 hours stoppage. During this time no circuits, vessels and associated rotary equipments are kept untouched or unattended from cleaning and inspections. Major to minor maintenance requirements are fulfilled with proper planning.
- Reaction shut down is planned every 30 days interval for preventive cleaning of gas circuits in scrubbers section and cleaning of high flow rate slurry circulation pumps' suction nozzles to maintain its performance unchanged.
- Selections of materials and its durability for various other equipment's and lines are noted satisfactory as compared with bench mark study.

4.1 Reaction System Details

The reactor is the heart of the process, so the most emphasis is placed on improving reactor performance. **Figure 1** shows the process flow diagram of PA. Our 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 19- 21% P_2O_5 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 with the pump capacity of 1800 m^3/hr, 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 98-100 °C. 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.

4.2 Reactor Volume Adequacy Study

Hydro over-sized the reactors to provide extra surface area to handle foam. These specified 2 m^3 reactor volumes per t/d P_2O_5 capacity x 700 t/d —> 1400 m^3. The original equipment list shows all three reactor have ID of 10,730 mm; WH (working height): 5,500 mm; TH (total height): 6,500 mm; working volume: 465 m^3 (which agrees with calculation of ID & WH).

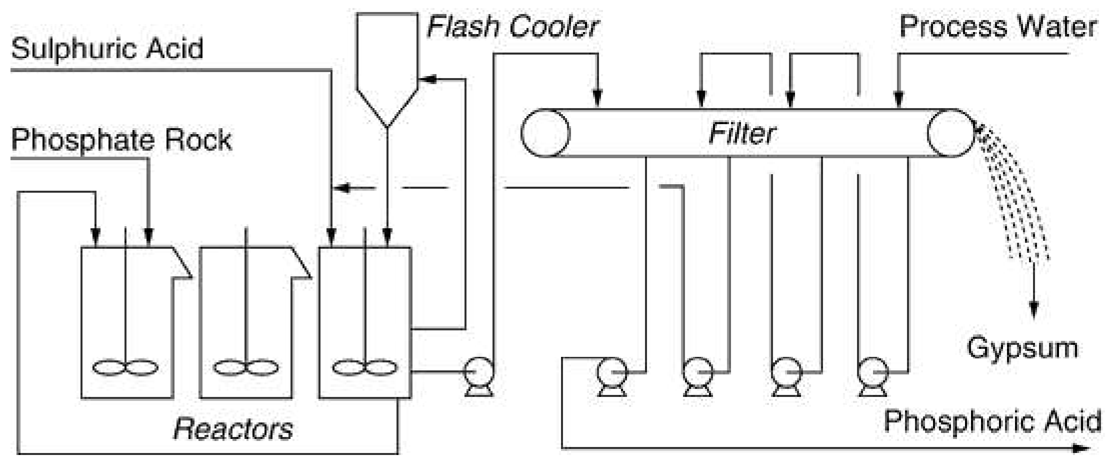

Figure 1. Process flow diagram of HH phosphoric acid

Table 3. Reactor dimension Comparison benchmark

Description	Comparative data			
	IJC	**Plant 1***	**Plant 2***	**Plant 3***
Plant load (t. P_2O_5)	700	750	1460	600
Reactor (M^3)	400*3	218*5	735*4	400*3
Reaction volume (Design) - M^3/MT of PA	**1.72**	**1.45**	**2.01**	**2**
Reaction volume(Working) - M^3/MT of PA	**1.42**			

** Plant 1, 2 & 3 are the reputed phosphoric acid producers:*

Table 4. Main technological and economical process parameter of IJC – phosphoric acid plant

Index	Unit	Values	Remarks
Rock Blending ratio	-	60:20:20	Additive rock, blended
Specific volume of reactor used on 900 MT production/day	M^3/TP_2O_5	1.3	More than 1 is acceptable
Degree of decomposition – rock phosphates	%	95.5 max	Due to +2 mm~6-7%
Gypsum washing efficiency	%	98.5	IJC needs to improve further on crystal size
Specific capacity of filter	T/M^2/day	5-5.2	Based on max production achieved (900 MT/day)
Specific consumption - RP	T/T	3.8-3.9	Due to +2 mm~6-7%
Sulphuric acid	T/T	2.75-2.86	Target3.1
Defoamer	Kg/T	9-10	Target<10
Anti-scale agent	Kg/T	2.5-2.5	Target <3.5
Raw water	M^3/T	3.5-4	Target<4.5
Power	KWH/T	180-190	Target 220

Three reactors at 465 m^3 equal 1395 m^3. 1395 m^3 / 700 tpd = 1.99. During normal operation there will be about 100 m^3 of free space in reactor 2, leaving an effective volume close to 1300 m^3.

Based on the comparison of other plant reactor volume data **(Table 3)**, IJC's calculated specific volume to run the plant at 110% load is 1.4 m^3 /tpd P_2O_5. Normally Hydro would provide about 1.4 -1.5 m^3/ tpd. On other projects, we have found that specific volume as low as 1 m^3/tpd is acceptable with Prayon multi-tank reaction systems (*i.e* more than 3 tanks).

However those Prayon multi-tank reactors have 8 or 9 compartments. The larger number of compartments provides more effective use of the total volume, compared to a 3-reactor system.

A specific volume of 1 m^3/tpd has not been demonstrated for a 3-reactor system like at IJC.

Main technological and economical process parameters of IJC-phosphoric acid are given in **Table 4**.

4.3 Protection from Large Size Particles Entry into the Reactor

Rock phosphate, received from JPMC mining area, sometimes is contaminated with big size bolder (size.>1").

That on passage to reactor creates huge nuisance to equipment and deposits at the bottom to reactor.

It not only reduces the effective reaction volume but also affects the agitator performance.

IJC took required steps to reduce big size stones from mines area by earmarking area for IJC and additionally installing 2- stages vibrating screen at assigned position on rock feed system to eliminate higher size particles. As the blended rock phosphate contacting average 9-10% moisture, installation of vibration screen mesh <10 mm was found to be not suitable as screen getting chocked. However, dry rock having 2-3% moisture once pass through the screen can eliminate desired limit of +4 mm oversize particles.

The small amount of oversize (+2 mm dry rock and +10 to 20 mm damp rock) from both the dry screen and the piano wire screen could be discarded.

However , in our cases particle size more than +4 mm was found to be 6-7 % in series of samples taken daily. The management wanted to eliminate such particles

Figure 1. An image of vibrating screen on-line in rock feeding system

by installation of an on-line crusher after vibrating screens **(Figure 1)** but not found to be economical.

4.4 Reactor Agitator

The blended rock used (additive rock) has about 80% of the feed less than 1mm size while the plant is designed for 80% less than 0.5 mm size. The coarse material above 2 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 10-20 minutes otherwise the carbon brick lining inside the reactor and propeller of agitator gets eroded fast.

The drained liquid is strained through coarse strainers to a sump from which the filtrate is recovered back to the reactors **(Figure 2)**. Installation of suction strainer to high flow slurry pump associated with 3rd reactor is given in **Figure 3.**

4.5 Scaling Issues

The solid content in the reactor slurry is increased by 1% to handle the additional quantity of solids generated on account of processing lower grade rock. Increase in scaling inside the flash cooler and slurry inlet and outlet ducts was noticed. There is a slight increase in the whitish silica deposits in the scrubbers and vapour ducts. The quantity of gypsum handled also increases from 5 tonnes to 6 tonnes per tonne of P_2O_5 due to increase of impurity percentage in gypsum from 8% to 10% due additional silica loading from input rock quality.

4.6 Filtration Efficiency

Normally in HH process gypsum loss is higher than the other processes because of its single filtration method. In IJC design, gypsum loss is 1.15 % P_2O_5 as insoluble and 0.50 % as water soluble P_2O_5 at 700 T P_2O_5/day load. We operate the plant more than 15 % higher than the design load and maintain the gypsum loss slightly higher than the design norms. Though, we are consuming the blended rock as feed, plant is operating now with overall efficiency of 88-89% and maintain the filtration efficiency as 90-91%. Gypsum crystals in majority are as agglomerated rhombohedral and approximately 20-25% finer size crystal and 2-5% are needle shape.

4.7 Zero Break-down Maintenance

IJC customized its maintenance practices based on its equipment failure rate and nature of failure. It has developed predictive and proactive maintenance practices for all its critical equipments. Routine maintenance practices are in line and routine cleaning maintenance schedule is strictly followed.

Upgradation of equipment and selection improved MOC for various parts of the equipment based on their failure rate are a cultural part of IJC's maintenance practices.

Figure 2. Reactor draining facility to remove settled heavier particles from vessels bottom

Figure 3. Installation of suction strainer to high flow slurry pump associated with 3rd reactor

IJC uses all spare parts and consumable items from most recognized and well versed manufacturing houses without compromising on the quality. Results are being recorded by operation and noted to be satisfactory. Last 3 years, no un-expected break-down or shutdown was experienced and 100% uptime was recorded. Complete survey of storage tanks in every three years and study of its health conditions is one of its best maintenance practices.

4.8 Gypsum Management

IJC follows dry disposal of gypsum and stacking methods. It consists of series of belt conveyors and projectile thrower system at the end point situated around 1.8 km away from plant site.

The filter cake at the exit of belt filters contains 18-22% w/w free moisture. Though filtration is normal but variation of moisture in the cake is noticed which is obviously due to the type of rock processed and the plant parameters.

Advantages

- Transportation of gypsum in dry mode is experienced simpler when compared to handling of gypsum slurry, which poses problems of pumping and plugging of pipelines.
- The initial cost for dry stacking is less compared to wet stacking, as the lining of the ponds and the complex under drain systems are not required.
- The problems of percolation and flooding of acidic water from the dry gypsum stacks are less in most of the cases, and virtually absent if the stacks are located in dry climate.
- The down-time of phosphoric acid plant is lower in this dry stacking.
- A separate maintenance team along with operational supervisor is continuously monitoring the gypsum and rock conveyors and necessary maintenance is done in time.

5.0 Concentrators and Cooling Towers Performances

IJC phosphoric acid cooling tower is forced draft type having six cells in line all time with one cell as spare. As per HH process designed goodness, the size of tower and capacity of pumps are considerably less than DH process.

Mass and heat load features in brief are as follows:

- Total 3000-3500 m^3/hour cooling water flow is sufficient to handle the heat load released from reaction section flash cooler, reactor gas scrubber, filter condensers & 3 numbers of concentrator for producing 850-900 MT/day production.
- Total hot water circulation flow to tower is equivalent to cooling water flow to process circuit for plant running.
- Differential temperature across the tower is 12-12.5 ^oC which is noted to be satisfactory.
- Phosphoric acid cooling tower performances are rated higher than as signed in 1990s. IJC has taken series of steps to control, monitor and correct its performances in day in and day out.

Following are the notable points of IJC maintenance practices

- Changing of cooling tower cell headers and drift eliminator twice in a week.
- Changing of pump seal from gland packing type to mechanical seal has given a good boost to be maintenance practices. The maintenance frequency has been reduced drastically owing to renewal of pump for gland leakage.
- Preventive maintenance of phosphoric acid cooling tower fans are taken every week interval as it encounters highly corrosive mist.
- In year 2014, phosphoric acid cooling tower inlet hot water trench route is channelized through settling pit, which settles the silica carried by water coming out from gas scrubbing section. This process improved phosphoric acid cooling tower performance to a large extent by reducing solid levels of cooling tower water.

5.1 Concentrator's Performance

IJC has three numbers of concentrator with capability to produce 1000 MTPD strong phosphoric acid of 54-56% concentration. IJC shares the customer satisfaction as its pride for producing premium quality acid.

- Life cycle for each evaporator at present stage is 10-12 days. Improvisation action plan is initiated for minimization of calendriya scale forming by introducing specialty chemicals prohibitive to avoid scale formation.
- IJC concentrators are well capable to produce 54-55 % acid in sustainable basis.
- IJC evaporators are capable to handle the designed performances :

o Specific LP steam consumption is 0.70-0.85 Mt/Mt P_2O_5 for 14-15% enrichment of concentration.

o Specific cooling water requirement concentrator is 3.5-3.7 m^3/Mt/hour P_2O_5 for getting heat load of 10 ^oC/Mt/hour.

o Capacity of each evaporator is 15 Mt/hr but capable to produce 18.5 Mt/hr. without affecting its quality.

A study was made on concentrators to reduce chlorides and fluorides and to increase efficiency in vacuum evaporative circuits operating under 100-110 mm bar vacuum. A series of study reveals that 80-85% fluorides and 55-60 % chlorides are eliminated from feed acid solution which are sufficient for SPA to meet the acid quality norms specified by international agencies for shipment.

6. Quality of IJC Merchant Grade Acid

Table 5 gives the quality of merchant grade acid produced by IJC.

Notable Features

- ♦ Fertilizer grades friendly, least effect on off grade generation on DAP.
- ♦ Enriched in P_2O_5 values un-parallel with many international suppliers.
- ♦ Lowest in Cl, F values & solid content.

6.1 Impurities and its Effect on Fertilizer Production and IJC's Efforts to Control the Minor Elements in its Production Unit

Fertilizer grade phosphoric acids produced by exporters are obliged to maintain the various minor elements presence to its below specified limit.

The boundary levels are further being narrowing down with a stipulated time limit for acid used for animal food grade customers.

Customization and tailor making product quality needs extra cost for processing. It is a challenge to the major phosphoric acid producers and IJC obliged the challenge and customized its product quality further as per requirement for food processing customers and opened up its market to European countries.

Customer feedback report reveals that IJC supplied phosphoric acid quality is superior to any other major producers and especially fertilizer grade acid has received very delightful customer survey report which enable IJC –delighted.

IJC has taken several trial runs in fertilizer unit within its group of companies to customize its quality standards and successfully eliminated the undesirable factor by a team of researchers. Following notable points are mentioned below:

• Magnesium (MgO) forms ammonium magnesium phosphates, Mg (NH_4) PO_4, as insoluble precipitate in DAP derivatives.

• RP MER is indicative of its suitability for its use in wet process acid production. Acid producers has to take sufficient precautionary measure on parameter controls and thus eliminate those elements to be best possible extent in reaction, filtration and storage acid

Table 5. Quality of IJC merchant grade phosphoric acid

Parameters	Unit	Values	Sample-I	Sample-II	Sample-III	Sample-IV
Specific gravity at 30^{o}c		**1.63 - 1.70**	1.674	1.678	1.677	1.669
P_2O_5	%	**52-56**	55.08	55.28	55.05	54.82
CaO	%	**0.05-0.5**	0.27	0.26	0.26	0.27
Chloride	%	**0.015-0.03**	0.011	0.012	0.017	0.018
Fe_2O_3	%	**0.6-1.14**	1.13	1.17	1.08	1.09
Al_2O_3	%	**0.5-1.2**	1.00	0.98	0.90	0.82
MgO	%	**0.2-0.6**	0.41	0.38	0.42	0.40
Fluoride	%	**0.3-0.4**	0.33	0.31	0.25	0.24
SO_4	%	**3 maximum**	1.89	1.90	1.93	2.24
O.M.	%	**0.03-0.1**	0.04	0.04	0.05	0.04
Solids	%	**0.2-1.0**	0.10	0.11	0.17	0.11

clarification stages.

The MER values formulated by notable scientist as:

MER = (Fe_2O_3%+Al_2O_3%+MgO%+MnO_4%)/ P_2O_5%.

MER <0.10, suspended of available solid in acid <2.0%.

- Manufacturing DAP and various derivatives of NPK will be hassle free as far as acid quality is concerned.

MER values marginally higher

- Affecting DAP processing can be adjusted by external addition of N by means of urea.
- It was experienced by our team that little adjustment of free sulphate level in feed acid and/ or controlling the conversions of MAP/DAP ratio in slurry feed to granulator.

MER values above the range 0.132

- DAP grades are failed and it goes as low as 16:48:0
- It needs to have a tough control N/P ratio in slurry fed to pipe reactor or pre neutralizer for further ammonization of slurry in granulator.
- N/P adjustment can be used to vary the Nitrogen and Phosphate level in the derivative 18:46:0 by adjusting the ratio of MAP (NH_4 H_2 PO_4) -lower N and higher P, and DAP [($NH_4)_2H$ PO_4]- for high nitrogen and lower P.

7.0 Sulphuric Acid Plant

7.1 Breakthrough Achievements in IJC- Sulphuric Acid Plant to Sustain the Highest Production

After achieving sustainable plant capacity utilization of phosphoric acid plant up to 120%, a parallel focus got shifted to sulphuric acid plant's improvement in overall capacity utilization that was 110 %. Total day production was marginally lagging to meet the sulphuric acid requirement in phosphoric acid plant.

Though sulphuric acid plant has crossed over its total production target with sustainable 110% capacity utilization but potentially it can be pushed to 120% production.

But it faced some insurmountable obstacle, as it seems, for further moving forward.

Sulphuric acid on-stream factor was achieved 98% but IJC targeted it to be 100 %.

Outsourcing of sulphuric acid from market was primary action plan and IJC started doing so for couple of months but it was not easy to match the economics of the business due to volatile S price in international market that makes the sulphuric acid procurement costlier and that to IJC could not catch the competitive market because of its location.

Basically sulphuric acid plant was facing frequently (at least once in a quarter) hot shut down due to:

- Tower acid circulation Pumps' discharge strainer blocking by black sticky mass **(Figure 4)** which started hindering flow to acid coolers after certain interval of plant operation.
- It makes an impact in pump tank acid temperature to be maintained high and high temperature of circulation acid will reduce the performance of absorption tower that leads to more SO_3 through stack.
- Due to which load cannot be increased more than 110 % and frequent hot shutdown was necessary to clean the strainers.

It was a great challenge to the IJC team to resolve the issue. The new journey started thereafter.

- Team visited different sulphuric acid producers in and around the country and shared their knowledge mutually.
- Invited designers and tried to understand the root cause.
- Frequently meet in SGD (small group discussion) and analyzed the probable reasons and step by step attempted to eliminate irrelevant causes.

Team concluded the following major contributors for black mass in sulphuric acid circulation line strainers.

- Lime used in sulphur- melting section for free acid neutralization.
- Erosion of ceramic in acid distribution bed in absorption tower.
- High silica content in process water for pump tank

Figure 4. Black sticky material was observed in the circulation acid system. Strainers were accumulated with this black material.

dilution

- Phosphate content in combustion air
- Oil content in dilution air for furnace view glasses

Based on many technical discussions and using statistical tools on PARETO analysis, it was finalized that high silica content in pump tank dilution water was the reason for this black material.

Then we installed the RO unit to reduce the process water silica content, and the RO water with less silica content has been lined up to DM as feed water.

We have installed the RO unit near to the WTP plant. RO water outlet was lined up to WTP as feed water, and then we improve the ion exchangers yield.

Result Obtained

- After lined up the DM water to pump tank, we observed that frequency of strainer chock has been drastically reduced to zero.
- Pump tank temperature was maintained at less 82 ^{o}C as per design.
- Plant running with 110% load consistently and possibility to move forward.

8.0. Way Forward

IJC is planning to run existing plant at 1000 P_2O_5 MTPD by utilizing the existing capacity in near future by adopting the following measures.

8.1 Installation of Additional Filter

As per operating procedure of HH process, the filter should be washed every 4 hours with hot water for every 7 days to avoid scale formation and 4 hours stopped for maintenance intention. Since we are operating two HH filters, we have to take filter wash two times in a week. During filter wash period, upstream of filter has been operating with 50% load and this leads to reduce the on-stream efficiency. By installation of third filter, we can operate the plant 100% continuously for the duration of filter wash time also.

We are making the modification in reaction system to increase the residence time. The IJC has decided to run the three filters continuously.

8.2 Agitator Design Modification

To avoid the solid settling in reactors and high feed rate, we are conducting the technical discussion with reputed agitator designers to change the agitator design in reactors. Presently agitator running with 110 KW hydraulic power, IJC is ready to increase the hydraulic power.

8.3 New Filter Feed Tank

The new seal compartment will be installed between reactor R2 and the filter feed tank. It would be approximately 2.5 m x 2.5 m x nearly 6 m deep, with its bottom sloping into the filter feed tank. A new opening would be cut into the upper side of reactor R2 to receive slurry from the new seal compartment. Ideally, the seal compartment would be close coupled to the filter feed tank on one side and to reactor R2 on the opposite side (like in Prayon mark 3 reactors).

The tops of the filter feed tank and seal compartment would be at the same elevation as the top of reactor R2. Consider either cylindrical or square shape of the new filter feed tank. A square tank requires no baffles.

Size of the slurry piping in and out of the flash cooler needs to be carefully evaluated.

8.4 Increase the Recirculation Flow

Recirculation from reactor 2 to 1A has to be proportional to production rate. This requires a bigger pump. The big change in rock quality might cause a need for a different ratio of slurry recirculation rate to production ratio to get optimum performance. This may require in-plant testing at different ratios. We are conducting the various studies to finalize the RC flow rate.

Conclusion

IJC got experience in sustaining the highest production in phosphoric acid plant and achieving the same by operating the plant higher than the name plate capacity. This has been done by utilizing inhouse expertise and with major capital investment.

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