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EXAMPLE OF REVAMPING PHOSPHORIC ACID PLANTS TO NISSAN C-PROCESS

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1. PREFACE

Phosphate fertilizer manufacturers are suffering from demand decrease and low price. In order to survive in such circumstances, all are paying best efforts for the reduction of production cost and the simplest way for it is cutting down of raw material and utility consumption.

In case of phosphoric acid production, main reduction methods of consumption are increase of P2O5 recovery from phosphate rock, saving of steam for concentration obtaining stronger acid from acidulation plant and use of this steam for co-generation and saving of electricity for rock grinding and agitation. The increase of P_2O_5 recovery can be achieved by incorporating recrystallization step into rock decomposition procedure and the stronger acid can be separated from hemihydrate calcium sulphate.

Nissan Chemical has various processes to comply with such requirements and here examples of revamping old phosphoric acid plant to more economical ones will be introduced.

2. PRINCIPLE OF HEMI-DIHYDRATE PROCESS

As shown in the Nordengren diagram given as Fig. 1, in direct dihydrate process which is the most popular process worldwidely today, the whole decomposition of phosphate rock takes place at condition of low concentration and low temperature marked D where calciumsulphate dihydrate is stable. On the contrary, in case of hemi-dihydrate process with one filter (Nissan H-process), the rock is decomposed under the condition of H_1 and the slurry is cooled down to H_2 to recrystallize the calcium sulphate to dihydrate achieving high P_2O_5 recovery of 97 - 98 % with good quality gypsum.

Straight hemihydrate process in which the rock is decomposed at the condition of C_1 and obtained slurry is separated into the product acid and hemihydrate gives strong product acid of 40 - 50 % but lower P_2O_5 recovery as there is no recrystallization step.

When separated hemihydrate cake in straight hemihydrate process is repulped again into dilute acid and recrystallized to dihydrate under the condition of C2 and this slurry is again filtered, both high product acid strength and high P_2O_5 recovery can be achieved. Nissan C-process belongs to this hemi-dihydrate process with two filters.

3. PROCESS FLOW OF NISSAN C-PROCESS

As shown in Fig. 2, this process consists of two sections, i.e., digestion section and hydration section. In digestion section, coarse phosphate rock of 9 Tyler mesh all pass is decomposed by sulphuric acid and return acid at the temperature of 90 - 100 °C. Two digesters are necessary and by controlling the sulphuric acid distribution of each digesters, high decomposition rate and easy to filter hemihydrate crystal can be obtained.

Phosphoric acid strength depends on the water content of raw material sulphuric acid and phosphate rock and requirements of fertilizer process to use this product acid, but it is generally 36 - 50 % P_2O_5. The slurry from the second digester is sent to the hemi-filter and the first filtrate is the product acid. The cake is washed by the first filtrate of the di-filter and this filtrate becomes return acid mixed with a part of the product acid.

Obtained hemihydrate cake is repulped by the second filtrate of the di-filter and sent to hydration tank. Number of hydration tank is not necessarily two depending on the hydration characteristics specific to raw material phosphate rock. The hemihydrate crystal is hydrated to dihydrate under mild agitation at 50 - 65 °C. Obtained good quality gypsum is filtered at di-filter and washed by water.

4. DESIGN PARAMETERS OF C-PROCESS

When a phosphoric acid plant is converted to a C-process plant, the reactor volume and the filter area are two main factors to decide the plant capacity. Necessary figures of volume and area are greatly different depending on the characteristics of the raw material rock, but quite vaguely expressed as follows:

Digester volume	1.2 - 1.8	$m^3/t\ P_2O_5 \cdot d$	(4 - 6 hours)
Hydration tank volume	0.4 - 2.2	$m^3/t\ P_2O_5 \cdot d$	(1 - 6 hours)
Hemi-filter area	5 - 8	$t\ P_2O_5/m^2 \cdot d$	
Di-filter area	7 - 10	$t\ P_2O_5/m^2 \cdot d$	

Generally rock decomposition rate of higher than 99 % and P_2O_5 recovery rate of 98 - 99 % are expected.

C-process needs two filters, hemi and di reaction section and digesters must be split into two. Consequently in most cases, installation of new filter is required. From here on, the examples of revamping existing phosphoric acid plants to Nissan C-process or others are explained for the cases of already completed, under construction and seriously studied.

5. CASE 1. NEW INSTALLATION OF COMPLETE HEMI SECTION

In most cases of revamping an existing plant, it is required that the plant shutdown period is minimized and the increase of the plant capacity is maximized. In such cases, complete hemihydrate section, i.e., two digesters, cooling system, pumps and hemi-filter are newly installed completely independent of existing plant and existing plant is utilized as dihydrate section. Only alterations of the existing plant are piping connection of di-filter filtrate line to hemihydrate section and slurry charge line to existing reactor and such work will be done in two weeks or so.

The capacity of the new plant will be decided by the size of existing filter which is used as a di-filter. Generally filterability of the dihydrate crystal in C-process is better than that in other processes and the capacity of the new plant can be increased by the ratio of filterability.

The rock grinding section can be completely bypassed in some cases but in most cases the rock is first sieved by about 2 mm screen and only over screen is ground.

The economic advantages of adopting this modification are

saving of steam for concentration	1.2 – 1.8 t steam/t P_2O_5
saving of electricity for grinding, etc.	30 – 60 KWH/t P_2O_5
increase of P_2O_5 recovery	1 – 5 %

and decrease of fixed cost due to capacity increase.

In general cases saved steam is used for cogeneration. The actual examples are shown as No. 1 to 5 in Table-1 attached.

6. CASE 2. ONLY HEMI-FILTER IS NEWLY INSTALLED.

Some processes adopt multi tank system. As the necessary volume of hydration tank in C-process is rather small, multiple tank plant can be converted to C-process by only adding hemi-filter utilizing existing some of the reactors for digesters and others for hydration tanks. In this case, agitation of digesters must be made stronger than that of hydration tanks and shutdown period for modification may be a little longer than that of Case 1. as the change of agitator drive takes some time.

The examples No. 6, 7, 8 and 9 in Table-1 belong to this case. In No. 6, a partition was installed in one of existing hydration tanks and used as digester-1 and 2. The existing evaporator of concentration section was used as flash cooler and only hemi-filter and three kinds of pumps were newly installed. In No. 7, as the raw material rock is special and longer retention time is required, digester 1 and flash cooler were installed besides hemi-filter. In both No. 8 and 9, no reactor was newly added, air cooling was reinforced and pumps and hemi-filter were newly installed.

7. CASE 3. COMBINATION OF TWO PLANTS

There are some cases where old two or more plants with plural number of vessels and filters are existing and production capacity decrease is admitted. In such case, the plants can be converted to C-process without adding any equipment.

Example No. 10 is the case and in this case the product acid is not very strong due to dilute raw material sulphuric acid similar to example No. 9. As this case is conversion from H-process, the advantage is only a certain steam saving but enough advantage is enjoyed due to little investment cost for conversion.

8. CASE 4. CONVERSION TO H-PROCESS

This case may not suit the title of this paper, but such case also exists as only high P_2O_5 recovery is expected by converting direct dihydrate process to Nissan H-process. When there is no use of steam and cogeneration is not acceptable, direct dihydrate process plant can be converted to H-process to obtain 3 - 4 % increase of P_2O_5 recovery. Example No. 11 is the case and several sections of dihydrate reactor were used as high temperature digestion section and remaining sections were used as low temperature hydration section. One hydration tank was added and cooling system was modified.

9. CASE 5. CONVERSION TO STRAIGHT HEMIHYDRATE PROCESS

When higher P_2O_5 recovery is not required and steam saving and production capacity increase are wanted, conversion to straight hemihydrate process is considered. In straight hemihydrate process, P_2O_5 recovery remains at 90 - 95 % but 40 - 50 % product acid can be obtained similar to C-process. In addition to that, as the filterability at straight hemihydrate process is larger than that at direct dihydrate process, the production capacity can be increased. Example No. 12 is the case and reactor of direct dihydrate process is divided into two sections, i.e., digester 1 and digester 2, and operation temperature is made higher. Investment cost is very small in this case, too.

10. CONCLUSION

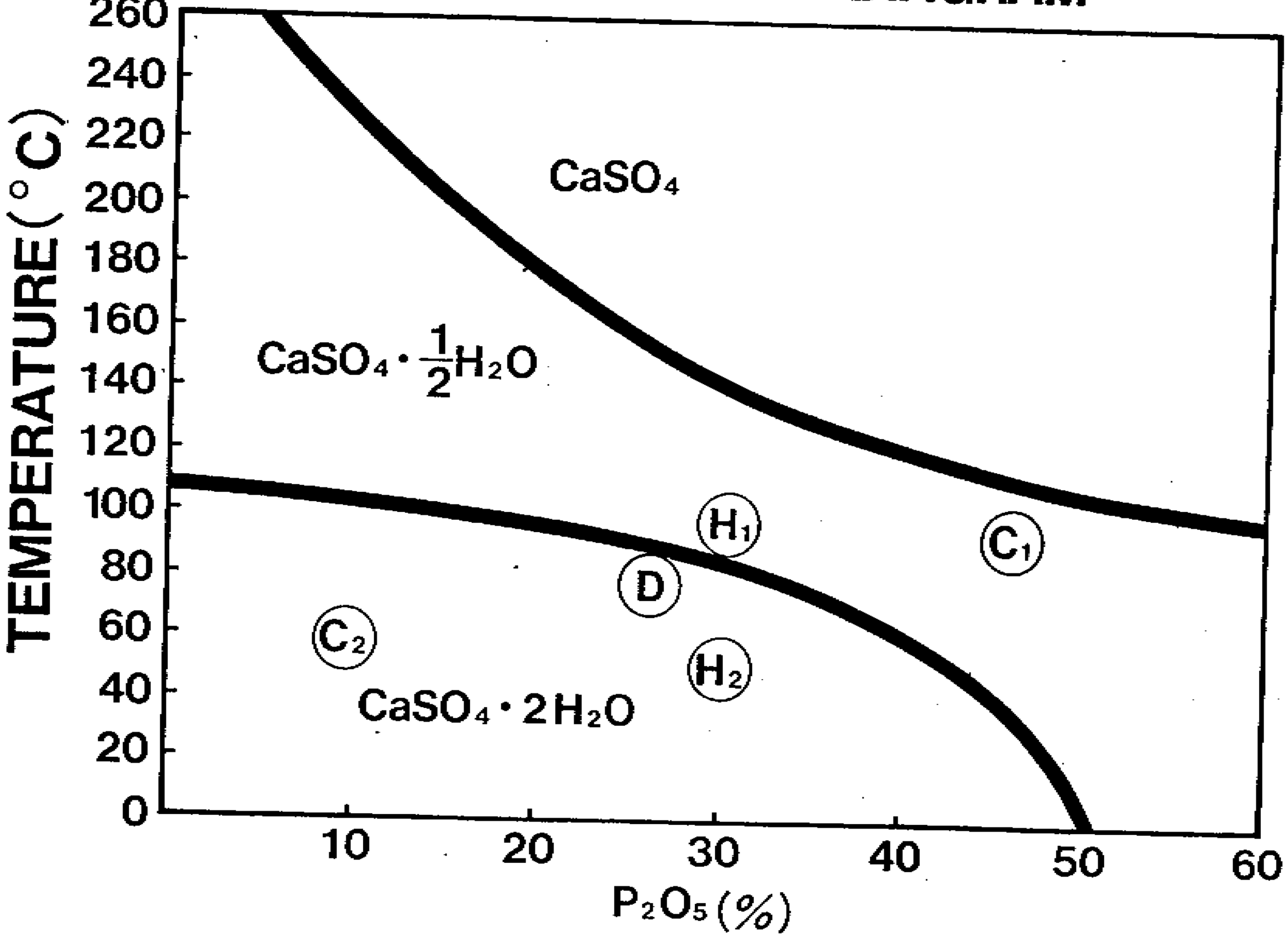
There are many possibilities of revamping existing phosphoric acid plants to more efficient ones by adopting hemihydrate process, but their actual ways differ very much depending on conditions of each factory and requirements of each factory. It will be our great pleasure if above-mentioned examples can be of some help for the study of modernization of your plants.

Table 1. Example of Revamping Phosphoric Acid Plants

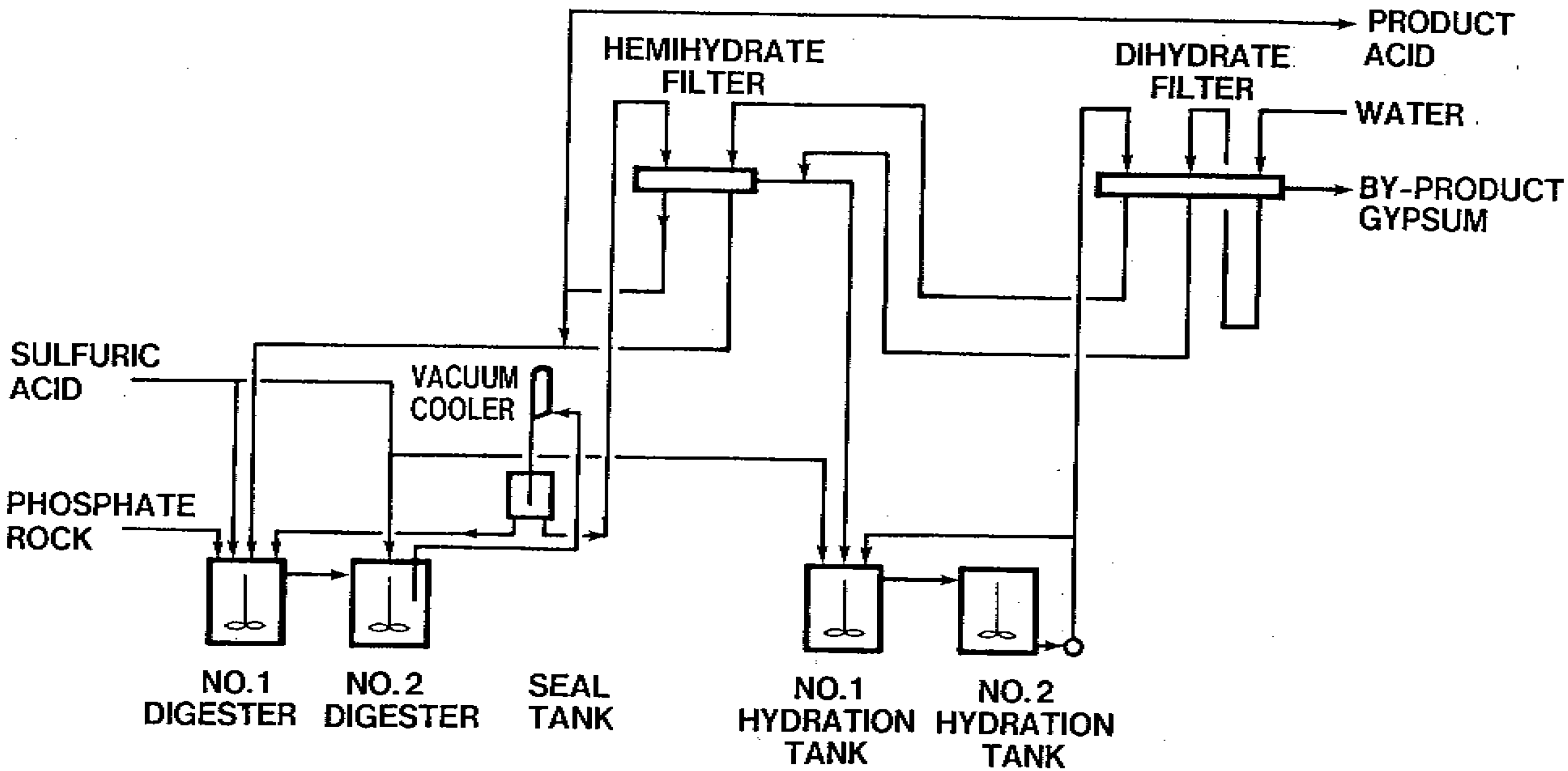
	Existing plant			Converted plant			New installation			Condition		
	Capacity T P2O5/D	Process	Filter	Capacity T P2O5/D	Process	Product P2O5 %	Hemi-Filter	Reactor	Cooling	Rock	H2SO4 %	Status
1.	60	H	belt	100	C	45	pan	2 x DG	air	Florida	98	O
2.	300	Direct di	pan	400	C	45	belt	2 x DG	flash	Florida	98	C
3.	300	Direct di	pan	450	C	45	belt	2 x DG	flash	Phalaborwa	98	S
4.	170	H	pan	220	C	45	belt	2 x DG	flash	Florida	98	S
5.	50	Direct di	pan	100	C	45	belt	2 x DG	flash	Yungnan	98	S
6.	230	H	pan	230	C	45	belt	-	-	Florida	98	C
7.	300	H	table	450	C	45	belt	1 x DG	flash	Fosfago	98	C
8.	500	H	table	600	C	45	belt	-	-	Morocco	98	S
9.	50+80	Hemi di.	pan & belt	90	C	37	belt	-	-	Florida	70	O
10.	130	H	pan & belt	70	C	38	-	-	-	Florida Morocco	72	O
11.	600	Direct di	pan	600	H	30	-	1 x HT	-	Florida	72	S
12.	300	Direct di	pan	375	S	45	-	-	-	Morocco	98	S

Notes:
- DG: Digester
- HT: Hydration tank
- O : In operation
- C : Under construction
- S : Under study

NORDENGREN PHASE DIAGRAM



SCHEMATIC FLOW DIAGRAM OF NISSAN C-PROCEES



TA/86/4 Example of revamping phosphoric acid plants to Nissan C-process by M. Miyamoto & Y. Yamanaka, Nissan Chemical Industries Ltd, Japan

DISCUSSION : (Rapporteurs Messrs M. Barloy, SCPA, France & A. HAMDI, SAEPA, Tunisia)

Q - Mr. N. KOLMEIJER, Windmill, Netherlands

Nissan have been publishing on several occasions about their processes, but as far as I know not how these were developed. Could you tell a bit more about this?
Do you have pilot plants or were the developments achieved by experiments on existing commercial plants?

A - I will answer for the case of Nissan-C process which was started to develop some 15 years ago.

First we made laboratory tests including bench scale tests and then went into pilot plant tests of about 1 t/d. After that a demonstration plant of 30 t/d was erected and operated for about one year.

Based on the results of this plant, a 100 t/d commercial plant was built. These days, we have accumulated enough experience and we do only bench scale tests with new rock and such rock can be applied directly to a commercial plant.

Q - Mr. A. BOURGOT, Prayon-Rupel, Belgium

a) Is it possible to know the reasons or the parameters for which digestion and hydration volumes are extreme?

b) How important is the influence of particle size of phosphate rock on the reaction volume for Nissan-C process?

A - a) Digestion volume does not differ so much in relation to the kind of rock. Its range of 1.2-1.8 m3/t/P2O5/d depends on the reactivity of the rock. Generally speaking hard igneous rock takes longer. Hydration time required differs very much depending on the rock. Igneous rock with high Si content like Kola rock takes very long for hydration.

b) We have set 2 mm as maximum rock particle size from our experience because we are afraid that particles bigger than 2 mm may settle at the bottom of the digester under the agitation condition we are now giving. It means that particle size is not an important factor for performance of the plant.

Q - Mr. L. RAHOUI, SAEPA, Tunisia

What are the performances of the Nissan-C process for a low grade phosphate rock, 26-28% P2O5 (acid strength, efficiency, filtration rates and reaction volume)?

A - Performances of C-process plant such as yield, tank volume, filter area, product strength, etc... are not so much influenced by BPL of rock itself but more by kind of impurities present in the rock because generally low BPL rock contains various impurities. Filtration rate will be lower in case of low BPL rock but other points cannot be commented simply.

Q - Mr. S. SAIDI, ICM, Tunisia

How can you keep a temperature of 50-65° C in the hydration tanks?

A - Temperature of hydration tank is controlled by either vacuum cooler or air cooling to remove hydration heat and reaction heat.

Q - Mr. B. PAULSON, Royster, USA

Do you have any experience in piling gypsum from a Nissan-C plant as a means of disposal?

A - In all C-process plants now in operation all gypsum is utilized. But in a plant now under construction, gypsum is disposed of by pond system. We have some experience with H-process and the nature of gypsum from H-process is not so different from C-process.

Q - Mr. T. LAINTO, Kemira Oy, Finland

a) What are the main reasons for the good filtrability (t/P2O5/m2) in Nissan-H process compared with normal DH-process?

b) Is it possible to work with one digestion tank in Nissan-H process?

c) Is it possible to work in Nissan-H process with polyethylene piping and rubber lining in high temperatures compared with normal DH-process?

d) Have you tried hard magmatic phosphates like Kola in Nissan-C process? Results?

A - a) The filtrability of H-process is good because recrystallization takes place at low temperature which is favourable to crystallization almost independent from reaction.

b) Number of digesters in Nissan-H process can be one but C-process must have two digesters.

c) We have never used polyethylene but we have used polypropylene. Rubber lining is normal material.

d) We have tested Kola rock for about two weeks as trial. It can be treated but hydration time must be longer than other rocks. Filtration was good.

Q - Mr. J.D. CRERAR, Norsk Hydro Fertilizers Ltd, United Kingdom

I have some questions on experience with conversion from dihydrate to hemihydrate.

a) Has the number 2 plant in table 1 which was under construction started yet? If so, what production rate, acid strength and recovery is achieved? (If not, why such a long construction time)?

b) What is the capital cost a new hemihydrate reaction and filtration section as described in case 1 (page 3)?

c) What practical experience does Nissan have in single stage hemihydrate process?

d) What are the free SO4 levels in N° 1 and N° 2 digesters (C-process)?

A - a) That plant is in India and contract was made early this year but the government clearance is not yet granted.

b) Investment cost is roughly 2 million US $ in case of 100 t/d plant and 5 million US $ in case of 500 t/d plant.

c) Nissan has no experience of single stage hemihydrate process in commercial plant, but it is understood the same as digestion in section of C-process.

d) Free sulphuric acid in N° 1 digester is in deficit. Ca content is between 0.5-3%, N°2 digester contains 2 - 5% H2SO4.

Q - Mr. Y. LOUIZI, SIAPE, Tunisia

a) Energy saving in grinding 30-60 kwh/t P205
b) Improvement of recovery: 1-5%. Why such high ranges?

A - a) Main electricity saving comes from saving in the grinding section. Difference from direct dihydrate process will be about 30 kw and from H-process will be 60 kw.

b) P205 recovery of C-process is about 98-99% and that of direct dihydrate process is assumed to be 93-96% and that of H-process is 97-98%.

Q - Mr. N. HUMMADI, JPMC, Jordan

What changes are needed to convert from DH to HDH, other than the reaction an filtration sections?

A - Reactor volume and filter area are two major points to decide plant capacity in case of converting direct DH plant to HDH process plant. Other points to be taken into consideration in conversion are materials of construction and cooling system.

Especially metal material of agitators pumps and filter must be carefully checked because both acid strength and temperature become higher. Cooling system must be modified in all cases but mainly by change of piping system. Degree of agitation also needs to be checked but generally it is not necessary to change it.