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PERFORMANCE AND OPERATION OF NEW NISSAN PHOSPHORIC ACID PROCESS PLANT

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1. PREFACE

Nissan Chemical Industries, Ltd. has developed a new process of producing wet phosphoric acid of high concentration, 45 % P_2O_5 or more, along the route of combination of hemihydrate and dihydrate steps, and has been running a small unit of commercial scale of 40 MTPD in its Toyama Works since the middle of 1974. Nissan made a presentation of paper regarding the performance and operational experience in this plant in 1975 in the technical conference of ISMA/ANDA held in Sao Paulo under the title of "Practice of New Nissan Phosphoric Acid Process". A new unit of 100 MTPD P_2O_5 was built for Rasa Industries Co., Ltd., who has been engaged in phosphatic fertilizers production for quite a long time and one of the leading fertilizer manufacturers in Japan, at its proprietary site Miyako, located in northern part of her main island, by Nissan Engineering, Ltd. in 1975. This plant was commissioned successfully in a short period and brought into a routine production. Here we are going to give a short description on the performance of this plant which well represents the characteristics of the New Nissan Process.

In Japan all phosphate rock is imported and almost all the phosphatic fertilizer manufacturers are destined to consider the use of various types of phosphate rocks, time to time available which could be dictated by the price, quality, stable supply and the other point of view, and such rocks might be used straight or blended with other rock of one or more sources. In this connection it would normally be required in a wet phosphoric acid plant design criteria that a flexibility and versatility of accomodating several rocks in the unit is to be taken into account. This case has not been exceptional from such requirements.

Rasa Industries Co., Ltd. has broad activities in copper and other nonferrous metals smelting and produces contact sulphuric acid and tower acid as well. Accordingly the strength of sulphuric acid could vary from monohydrate to 67 %. This should have been taken into account in this plant.

Furthermore Rasa Industries Co., Ltd. has started production of ammonium polyphosphates, as a pioneer in this scope in Japan, which are being realized to be most efficient phosphate as fertilizers, and it requires the primary raw material phosphoric acid be as strong as possible. The product acid from the new plant is more meeting to such requirement than the acid produced in the conventional processes.

2. PROCESS FEATURES

Here we would remind the features of New Nissan Phosphoric Acid Process as follows:

(1) Phosphoric acid of high concentration can be produced directly without evaporation - The concentration reaches 45 to 50 % P_2O_5 if there is not any particular restricting factor.

(2) P_2O_5 recovery is high - Despite of high product acid concentration a high P_2O_5 extraction efficiency is achieved comparable to the recovery figure of 97 % in the conventional Nissan Process or higher.

(3) Gypsum of supreme quality which is suitable in the use for plaster, wallboard and cement retarder can be obtained. - Good physical characteristics of by-product gypsum have been one of the process features of Nissan Process and better qualitied gypsum could be expected in the New Process.

(4) Wide varieties of rocks are processed in high efficiency. - Igneous type rocks such as Kola can be processed as well as rocks of sedimentary origin.

(5) Coarse rock screened or only coarsely ground can be used. - This results in the saving of electric power required for rock grinding and elimination of rock grinding mill instalation.

(6) The product acid is free from a sludge. - A trouble of sludge formation during and after concentration of acid and of sludge removal is no longer a serious problem in obtaining

concentrated acid.

(7) Running cost of the process plant is remarkably low. - Substantial saving of utilities, electric power for grinding, steam for concentration, as well as elimination of large amount of cooling water and the decrease of raw materials consumption could contribute in this effect.

(8) Total plant investment cost is less expensive. - Investment cost savings resulting from elimination of acid evaporator, sludge removal thickener, lowering-down of mill capacity or its elimination and cutting-back of relevant auxiliaries are the major items to be counted in this connection.

3. OUTLINE OF THE PLANT

The Rasa Miyako Plant was designed and constructed on the experience of 40 MTPD commercial unit of Nissan Toyama. Two units of existing belt filters of boat bottom shaped construction were utilized for the dihydrate filtration service. A tilting pan type filter made by Mitsubishi Chemical Machinery Mfg. Co., Ltd. was installed for hemihydrate filtration. All the agitated reaction vessels are rubber lined construction with rubber lined single agitators. The simplified flow sheet of this plant is attached hereto. Also several pictures showing the parts of this plant are presented by the courtesy of RASA Industries Co., Ltd.

It was indicated that the plant would be run using Florida rock of various grades, Morocco rock and possibly others as well and also produce concentrated acid as strong as possible in future. Among them one of the most essential and important requirements was the quality of by-product gypsum which should be suitable for various uses. These were carefully studied and incorporated in the plant design.

Coarsely ground rock of 32 Tyler mesh pass is circulating all the time through rock circulation unit and a metered portion by aid of a belt weigher is brought into a premixer, where a metered flow of return acid of which strength is regulated strictly and also a metered amount of recycle hemihydrate slurry come together and wet the rock under strong agitation.

There are two digesters of different size, the first one is much bigger than the second. The majority, more than 90%, of stoichiometric amount of sulphuric acid is metered and mixed with required amount of return acid to bring down the strength of sulphuric acid to around 80% in the mixed acid and then cooled in a vertical shell and tube heat exchanger. This mixed acid is proportionally fed into two digesters, about 50% into the first and the balance into the second. This proportionment is quite important to obtain clustered hemihydrate crystals which dictates the filtrability of hemihydrate. A cooling tank and a small pumping tank are provided. The removal of reaction heat hence the cooling of hemihydrate slurry is mainly performed in this cooling tank with multiple pipe air spargers, air blown through just across the surface of slurry. The temperature of slurry is brought down from 90 °C to 75 °C. Gases from these reaction vessels are collected together and pass UA scrubber of slit contact type and fluorine compounds in the gas are washed almost completely. Hemihydrate slurry is pumped up by a slurry pump of horizontal centrifugal typo, casing and impeller of which are rubber covered, and distributed to a hemihydrate filter and to premixer. One wash is applied to hemihydrate filter cake with medium strength acid which comes from dihydrate filters as first filtrate. The cake is washed here fairly well and the washing efficiency ranges 80% to 90% depending to the physical characteristics of clustered hemihydrate. Second filtrate from hemihydrate filter thus obtained is collected in a return acid tank and with addition of product acid the strength of the return acid is maintained within certain narrow range namely around 38% P_2O_5. Flow rate of wash acid applied to hemihydrate filter is also regulated.

Hemihydrate filter cake is washed off the tilting pans of filter and repulped by aid of second filtrate from dihydrate filters. Hydration tanks are comprised of four tanks provided with mild agitation. Small quantity of sulphuric acid of balance amount is added into the top tank. Seed slurry from the last tank is recycled. Temperature in these tanks is regulated in the range of 50 to 60 °C so that the hydration proceeds under moderate conditions. Hemihydrate once dissolves in the acid and recrystallises giving uniformly sized good crystals and high rock decomposition percentage more than 99%.

Dihydrate slurry is pumped with a Warman Pump of the same construction as hemihydrate slurry pump. Slurry is evenly divided and fed on each of two belt filters. It is desired that the thickness of filter cake on these filters is uniform across the belt to obtain better filtration efficiency. Sometimes, however, it was not so easy because of the shape (cross section) of boat bottomed filter as gypsum gathered thick at the centre of the traveling belt leaving the thickness of cake on the edges of belt thinner.

First filtrate from both filters is sealed and collected in one tank wherethrough it is pumped to the hemihydrate filter as a wash acid under controlled flow rate. Dihydrate cake is washed with hot water and consequently gives second filtrate of weak acid. Second filtrate from both filters is sealed and collected in one tank in the same way as first filtrate and used for repulping of hemihydrate filter cake. The washing efficiency in dihydrate filtration reaches as high as 98%.

4. PLANT PERFORMANCES AND OPERATION

The erection work of the plant was completed in the middle of December, 1975. In two weeks period of the latter half of December minor plant adjustment was conducted coupled with the initial acid feed and starting-up of the plant during which such requirements for amendment and adjustment from operational point of view, as usually found in any plant start-up, were enumerated and advised to be effected. January, 1976 was completely a dead period for phosphoric acid production demand by Rasa Industries. The plant was warmed up and brought to start up on 4th February. Total working period allocated to this plant in view of the production programme of Rasa Industries could not last so long. Time for bringing the plant on stream was quite limited. Under the circumstances the plant was started up at 60% load on 4th February and gradually brought up. Short shutdowns of mechanical nature took place several times, however the plant commissioning was finished by 20th of February including successful demonstration period of plant performance guarantee of three days. A diagrammatic illustration of the plant start-up is attached. A smooth and trouble-free start-up as such implies a most reliable and high process stability based on the operation experience and technical knowledge of the foregoing 40 MTPD plant in Nissan Toyama.

In the start-up Florida rock of 72 BPL was fed into the plant of coarsely ground to 32 Tyler mesh (approximately 0.5 mm). The concentration of sulphuric acid was 96%. The strength of product acid was brought up to 45 - 46% P_2O_5 immediately after initial feed of raw materials as shown in attached figure. P_2O_5 recovery demonstrated was 97.7%.

The plant has been run on other rock blends since then.

Some detail of the performance data will be given in slides.

5. Quality of Products

5.1 The example analyses of acid, calcium sulphate hemihydrate and gypsum are illustrated in comparison with the Florida 72 BPL rock as raw material.

	Phosphate rock	Product acid	Hemihydrate	Gypsum
P_2O_5	32.11	45.49	1.56	0.43
Water Soluble P_2O_5				0.16
CaO	46.71	0.15	35.21	30.62
SO_3	1.04	3.60	50.46	44.09
F	3.87	1.30	1.30	0.56
SiO_2	6.40	0.41	3.65	3.57
MgO	0.29	0.39	0.01	0.00
Fe_2O_3	0.90	1.00	0.02	0.01
Al_2O_3	0.81	0.74	0.20	0.04
Na_2O	0.50	0.07	0.54	0.30
K_2O	0.26	0.08	0.18	0.16
CO_2	3.42			
Loss of Ignition	2.12		3.84	2.43
Combined Water				19.24
SrO	0.16			

The distribution of impurities in rock has been investigated and the same trend was observed in the distribution of various components with that, in the paper presented by us in ISMA Technical Meeting in São Paulo last year, which was calculated on North Carolina rock.

	Acid	Gypsum	Evolved
F	20	36	44
SiO_2	5	56	39
MgO	96	4	0
Fe_2O_3	99	1	0
Al_2O_3	80	20	0
Na_2O	8	92	0
K_2O	14	86	0
Organic Matter	58	42	0

5.2 By product gypsum quality was tested in the purpose of using for gypsum wall board and cement retarder. The quality was found to be satisfactory for those uses and rather better than the gypsum produced in Nissan conventional process. The comparison is given as follows.

For gypsum wall board use

	RASA	Conventional Nissan
Bulk Density (g/ml)	0.900	0.623
Consistency (%)	65.4	74.8
Setting Time (sec)		
Initial	349	357
Surface Hardening	469	509
Final	1651	1428
PH	6.0	5.8
Wet Tensile Strength (kg/cm^2)	18.8	10.0
Adhesion (%)	86	50

For cement retarder

	RASA	Conventional Nissan
Setting Time		
Initial (min)	215	225
Final (hr)	5.2	5.1
Stability	good	good
Compression Strength (kg/cm^2)		
3 days	139	128
7	223	215
28	396	382

5.3 Other Analyses

Scaling was not a significant problem by itself for the continual operation. Analyses of the scale collected from hemihydrate filter and from the dihydrate filter were made for comparison purpose. An example of such analyses is given hereunder.

	Hemihydrate	Dihydrate
Moisture	11.6	3.6
Total P_2O_5	0.76	11.22
CaO	16.00	1.27
SO_3	22.23	1.69
F	25.40	41.70
SiO_2	13.12	23.78
Al_2O_3	0.56	0.34
Na_2O	12.53	15.33
K_2O	0.59	23.31
Loss of Ignition	39.47	48.00

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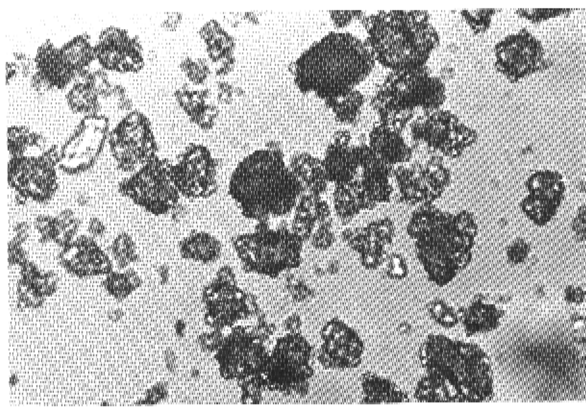
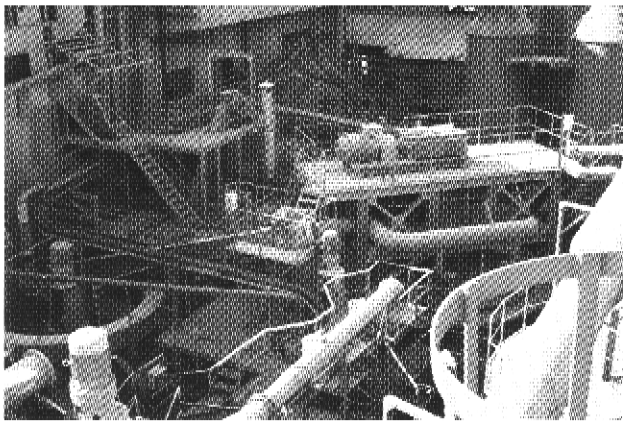
Microphotographs

Clustered Calcium Sulfate Hemihydrate

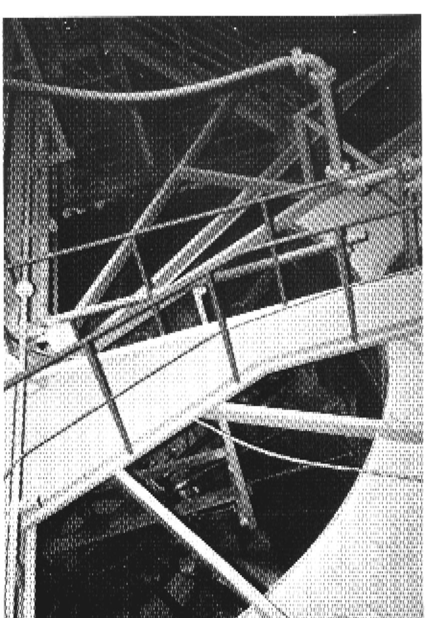


Calcium Sulfate Dihydrate

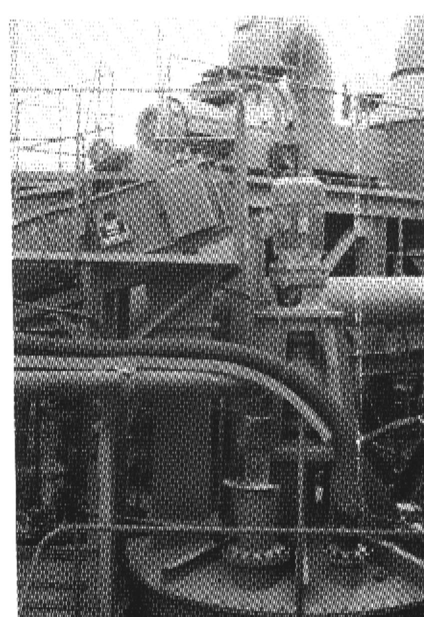
Hemihydrate Reactors



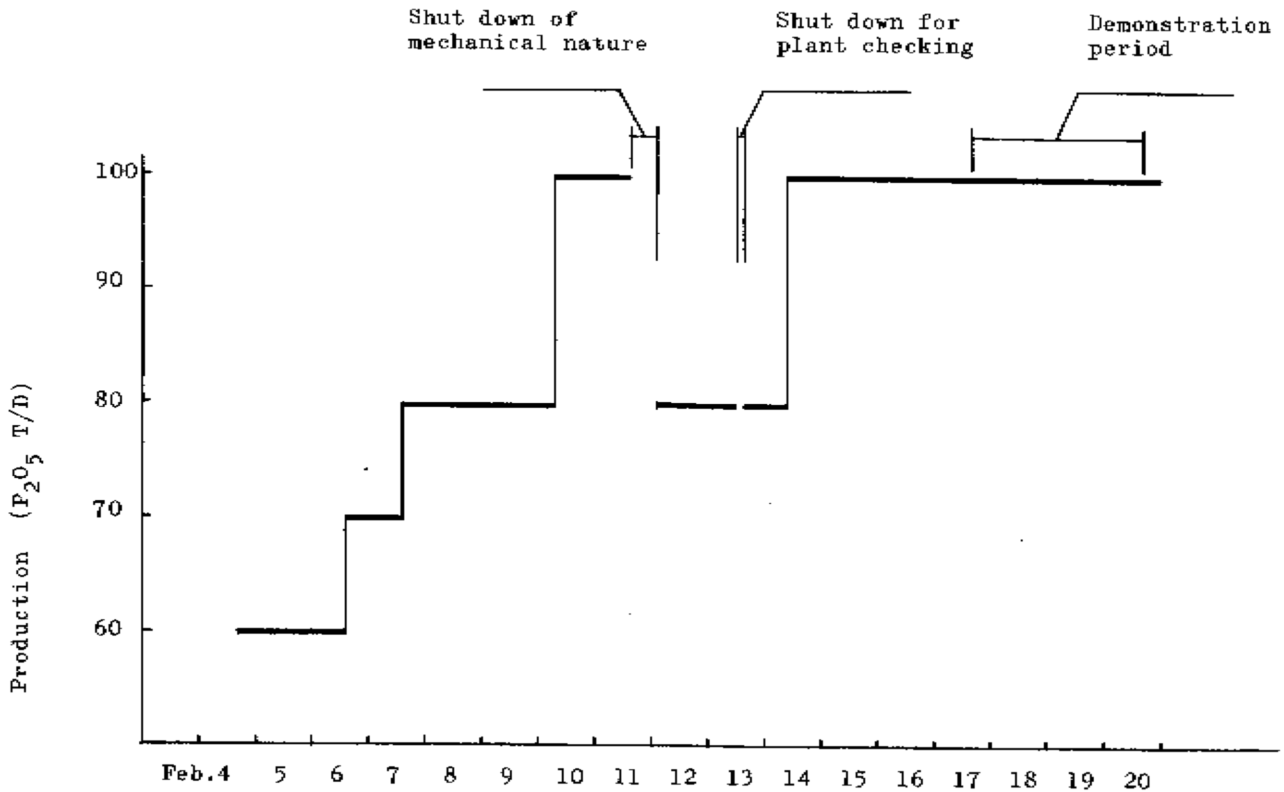
Centervalve of
Hemihydrate Filter



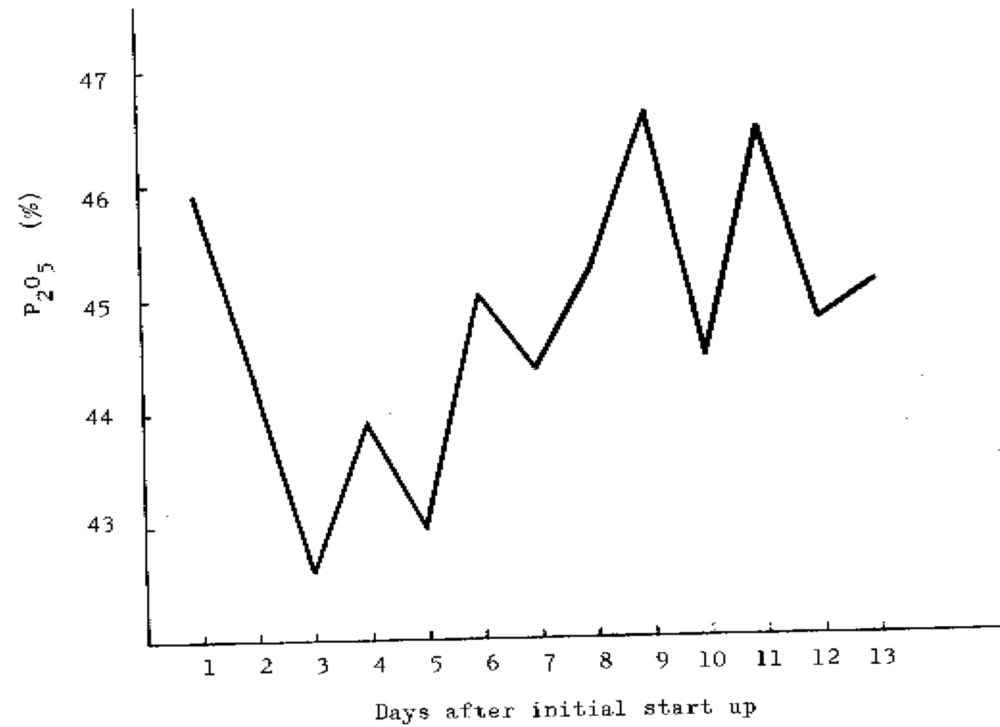
Top of Premixer

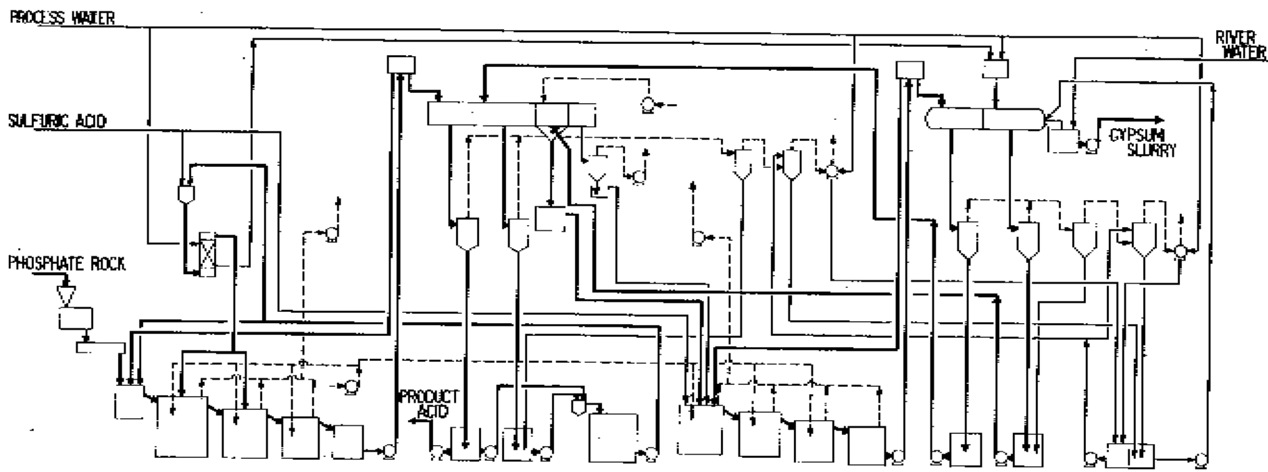


Diagramatic illustration of the plant start up



P_2O_5 Concentration of Product Acid





PROCESS FLOW SHEET