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NEW SULPHURIC ACID INSTALLATION OF "SOCIETE BOZEL-MALETRA" at PETIT-QUEVILLY (S. Inf.).

by P. Flament.

For a little more than 20 years the sulphuric acid market has shown two very distinct tendencies which are at the same time the effect and cause of progress. The reactions of Sulfonation require concentrated acids and the development of artificial textiles has only been possible on the basis of relatively pure acids. Hence the two tendencies: the continued increase both in the consumption of oleum and in increasingly concentrated acids and the necessity of an increasing purity. Only the contact process allows of obtaining simultaneously oleum, high concentrations in monohydrate and the quality.

Speaking about Europe in general, the rare occurrence of sulphur and the price resulting therefrom have induced industrialists to use sulphur-bearing minerals (pyrites, blendes, etc.). In spite of this, the sulphur in these minerals is still very dear and constitutes, in fact, more than 80% of the cost price of the acid produced. Improvements were carried out at all stages of manufacture with a view to obtaining an optimum output based on the total sulphur used.

It is from this angle that we propose briefly to describe the recent installations of "Societe BOZEL-MALETRA" at their works at Petit-Quevilly and to indicate the results obtained.

In addition to the research carried out in regard to optimum output, an aim equally aspired to was elasticity of operation, which enables the manufacturers to satisfy the demand of customers varying between 35 and 120 tons per day of contact acid or oleum. That is why instead of one single catalysor unit of a nominal capacity of 100 tons per day, two similar units with a capacity of 50 tons per day have been installed. Generally speaking, the other various apparatus are smaller but more numerous than in the case of a normal installation for 100 tons per day. The initial installation costs are certainly much heavier but elasticity and smooth running have been improved.

1. PRODUCTION OF SULFUROUS GAS

This production is effected by five large furnaces, three of the Maguin type, some several years old, with a capacity of 18 tons of pyrites per day, two of the Induschimie type with a multiple feeder which allows of a variation in capacity ranging from 16 to 30 tons of pyrites a day in extreme cases, the normal range being 19 to 27 tons per day.

It is the above reason which has dictated the use of those furnaces rather than the horizontal rotary furnaces or flash roasters.

It appears to us useful to give some supplementary information with regard to feeding and the construction of those furnaces.

In view of the fact that with furnaces having a single upper feeding unit, the quantity of pyrites burned per square metre of hearth suitable for roasting, amounts to between 80 and 100 kgs, this quantity can rise to 150 or even 170 kgs with the "Induschimie" multiple feed furnace.

Pyrites are introduced into the first three odd-numbered hearths (centri-petal hearths). The proportions introduced into each hearth can be regulated according to the total charge of the furnace, the quality of pyrites and the rate of combustion. It is possible, for example, to supply to hearth 1, 50%, hearth 3, 35% and hearth 5, 15%. It is a priori evident that the temperature is thus regulated and that, at the same time, the active combustion zone is spread over a greater number of hearths than in the same furnace fed to the extent of 100% in the upper hearth.

In addition to these advantages, there is a better equilibrium between the expansion and cooling of the mechanical parts.

These furnaces are very elastic and permit of the roasting of flotation pyrites (such as Cyprus and Boliden) the combustion rate of which is very considerable and which are dangerous to normal furnaces. In the case of a stoppage of one of them, it is, on the other hand, possible with a battery of 4 or 5 furnaces, to avoid a falling-off in output by a rapid increase in the actual charge of the furnaces remaining in operation.

With a pyrites such as the Kalavassos with 48% of sulphur the sulphur burning efficiency is 98% minimum.

As to construction, it has been known for more than 10 years, that the lining and the hearths built of refractory materials have been replaced by some industrialists by a concrete made of fused Lafarge cement in the construction of the furnaces of a small diameter (up to 4.50 metres). At the present time, the same material is being used for furnaces with a diameter of 6 and even 6.50 metres.

The manufacture of refractory concrete certainly requires much care but it is possible to replace the specialists in furnace setting by reliable and conscientious masons. Byway of example, we would mention that the interior fittings of a Maguin furnace of 18 tons per day (6 metres in diameter) and comprising the walls and 10 ceilings have been built within 20 working days with 11 workmen, 4 of whom were masons. For the same furnace constructed with refractory bricks one would have to reckon with 65 working days with 7 workmen two of whom would be specialists in furnace setting.

The times for attaining the required temperature are also widely divergent. With concrete 8 days are sufficient to heat a mass of 168 tons saturated with water. With refractory bricks three weeks would be necessary so as to avoid rough treatment. Finally, the materials necessary only represent 2/3 of the price to be paid for fitting with shaped refractory material.

II. PURIFICATION OF SO_2 GAS.

This purification is effected by

(1) an electric dust precipitator of the Cottrell type in two installations, one constructed by SPIG, the other by CECA. We

should like to recall that the coolest points must have a temperature of more than 350° centigrade.

(2) an empty tower in which a very large quantity of acid is atomised and which has the function of cooling and de-arsenating the gases which leave the electric dust precipitator.

The circulation of acid absorbs As_2O_3 which has to be eliminated continuously at a well chosen point of the circuit. The necessity of cooling energetically large quantities of acid may, however, present difficulties.

A happy solution appears to have been found by "Société Induschimie". After concluding semi-technical trials, construction is actually being carried out and we propose to describe only the principle thereof. Crystallisation of As_2O_3 has to take place during movement and the velocity of the circulation of the acid in the crystalliser must be such that the crystalls remain fine and do not stick. By creating a circuit branching from the main circuit and by submitting the acid of this small circuit to energetic agitation and cooling it is possible to avoid a thorough cooling of the total quantity of the circulating acid in order to maintain an As_2O_3 content which is not inconvenient.

This empty tower recuperates the SO_3 formed in the furnaces and allows production of an acid with 50% monohydrate suitable for the manufacture of fertilisers after-concentration.

(3) a cooling tower the aim of which is to improve the purification of SO_2 and to decrease the temperature of the gases as much as possible and, in any case, below 30° centigrade.

(4) electrostatic precipitation of the mist in two units of the wet Cottrell type, one constructed by SPIG and the other by CECA.

The gas is then dried in a tower and the upper portion of which forms a screen so as to avoid the entrainment of acid mist in the stream of dry gas.

This drying permits gases with 7% SO_2 and containing less than 60 milligrammes of water per cubic metre to be obtained.

In order to limit the losses due to dissolution, in the drying acids, of SO_2 which would be liberated in the absorption tower, the circulation in the drying tower is independent from the circulation for absorption. The content is maintained by adding 98.5% acid and the minimum quantity of the weakened acid is recycled to the nearest absorption tower. This small quantity of acid which only depends upon the humidity of the gases, i.e. of their temperature, can be freed of gas at the outlet of the tower by an air jet. This supplementary air only slightly dilutes the gases travelling to the catalyser.

III. OXIDATION OF SO_2 GAS.

The installation comprises two catalyser units of the INDUSCHIMIE type, having 2 stages and an intermediary exchanger.

These apparatus are carefully protected against loss of heat which gives them a considerable heat capacity, hence an insensibility to exterior conditions and to stops of a short duration.

It is evident that the numerous possibilities of regulation make it possible to circulate relative proportions of hot and cold gases necessary to obtain a system of constant temperature.

It would have been simpler to construct an apparatus with a nominal capacity of 100 tons per day with three stages but we have already given the reason for our choice.

The Vanadium catalyser manufactured by Induschimie has been studied with particular care with a view to obtaining
- a very pronounced mechanical resistance and almost total insensibility to thermal disturbances

- a great activity which enables the manufacturer to obtain an optimum output with a minimum volume of catalyser.

- a low charge, hence an economy in driving power.

The dimensions of the grains of the catalyst very according to whether it is the first stage which works at a higher temperature and which contains the production mass (these give in fact 80 to 85% of the total conversion) or the second stage which represents the finishing mass or output and which, must ensure a final conversion of 98.5%.

In these two units of nominal 50 tons each, the quantities of masses are approximately 450 litres per ton produced per day. Those apparatus are extremely elastic and easy to run. It is possible to obtain all the rates of operation from 37 tons to 65 tons a day.

Without the nominal charge, the conversion attains 98.5% and with 60 tons it does not go below 97.7% with gases having a SO_2 content between 6.5 and 7%.

IV. ABSORPTION OF SO_3 PRODUCT

There are two towers which absorb more than 99.5% of the SO_3 produced.

The first tower, or Oleum tower, received the major portion of the gases at a temperature in the neighbourhood of 160° centigrade. It is sprayed with a considerable quantity of 20/25 oleum, the concentration of which is maintained by an addition of 98.5% acid.

The second, or 98% tower, receives the remainder of the SO_3 from the preceding tower together with a portion of the direct gases at 160° centigrade. It is sprayed with a considerable quantity of 98.5% acid, the concentration of which is maintained by the addition of an acid decreased to 97% coming from the drying tower or by the addition of water.

The circulating acids are energetically cooled in tubular coolers. A single absorption tower would suffice for the manufacture of 98% acid without oleum.

The use of immersed vertical pumps enormously simplifies the installations and avoids the trouble of installing stuffing-boxes. The acids obtained contain less than 1 milligramme of arsenic per kg and 30 to 35 milligrammes of iron.

The absorption is almost complete and no trace of SO_3 is noticeable in the chimney. This installation is very easy to run thanks to the apparatus for controlling the acid content, placed at the disposal of the staff.

V. CONTROL

The control is ensured by

(a) two electric SO_2 analysers of the Siemens type, well known and reliable,
(b) two multicurved MECI thermometric apparatus (Leeds and Northrup patents)
(c) two electronic Speedomax galvanometers of great precision

with which any deviation of the indicator is immediately visible. They make it possible to read off 25 temperatures in less than one minute.

(d) titrimeters and potentiometers for absorption acid which give a continuous measurement of the content of 98% tower acid, between 96 and 99% (the figure of 98% is in the centre of the diagram), as well as the content of oleum, between 16 and 25, of the other tower.

VI. RECAPITULATION OF OUTPUT

(1) Furnaces — Minimum output 98%

(2) Purification of SO_2 — Minimum output 99.5% (taking into account 4 to 5% reclaimed in the form of impure 50% acid and coming from the SO_3 of the furnaces)

(3) Drying — Minimum output 99.5% (with degassing of double circuit)

(4) Catalysis at a normal fluxion (attaining 99.6% of recuperation of sulphur with a bi-sulphite installation in front of chimney)

(5) Absorption — Minimum output 99.5%

Total output based on total sulphur introduced into furnaces: 95% minimum.
Consumption of Kws: 40 per ton of acid produced.

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