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**HISTORY OF FLUORINE RECOVERY PROCESSES**

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The aim of this presentation is to demonstrate the wealth of information developed during the past 50 years on the subject of fluorine recovery. In the first place we must analyse the various processes where the treatment of phosphates and phosphoric acid cause the evolution of fluorine and what success has been achieved in the reduction of fluorine evolution from these processes.

Thus the processes that will be studied are:

- **The Production of Single-superphosphate**
- **The Production of Triple-superphosphate**
- **The Production of Phosphoric Acid**
- **The Evaporation of Phosphoric Acid**
- **The Defluorination of Phosphoric Acid**

## 1 Some General Comments

Firstly however some of the characteristics of fluosilicic acid should be addressed. The chemical formula of Fluosilicic Acid is  $\text{H}_2\text{SiF}_6$ . However things are not as simple as that due to the fact that rarely is fluosilicic acid present as pure  $\text{H}_2\text{SiF}_6$ . In general the concentration of fluosilicic acid is determined and expressed by measuring the fluorine content and then expressing the fluorine content as  $\text{H}_2\text{SiF}_6$ . A rigorous analysis can be made by measuring the Fluorine and silica contents and expressing the result as  $\text{H}_2\text{SiF}_6$  and free HF. However this does not cover the case where the Silica is in excess of the stoichiometric ratio of  $\text{H}_2\text{SiF}_6$ . This phenomenon is well reported in several of the references quoted at the end of this presentation. There are well reported references to the existence of  $\text{H}_2\text{SiF}_6 \cdot \text{SiF}_4$ . This species, with a molar ratio of 5 F to 1 Si exists in fluosilicic acid recovered from the evaporation units during phosphoric acid concentration when the final acid strength is of the order of 48 - 54%  $\text{P}_2\text{O}_5$ . Hereon in this presentation, FSA means a mixture of HF,  $\text{H}_2\text{SiF}_6$  and  $\text{H}_2\text{SiF}_6 \cdot \text{SiF}_4$ .

The determination of HF and  $\text{H}_2\text{SiF}_6$  in a solution is effected by titrating the acid cold, cooled by ice, and hot. The cold titration measures the fluorine present as free F<sup>-</sup> whereas by heating the solution all the  $\text{H}_2\text{SiF}_6$  dissociates to F<sup>-</sup>. A longer method is the determination of F and  $\text{SiO}_2$  and the back calculation of the ratio of HF and  $\text{H}_2\text{SiF}_6$ .

It is this variation in the silica to fluorine ratio that makes the interpretation of known data so difficult as rarely do the texts clearly define the composition of the fluosilicic acid accurately. Thus the values and conclusions inherent in each of the presentations are difficult to interpret.

It is obvious that the silica to fluorine ratio in the phosphate or the acid produced from a specific phosphate are critical with respect to the quality of FSA produced in any specific case. The other point to be

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considered is the “reactive” silica and how this is determined. The analysis of “reactive” silica is normally a proprietary method that treats a phosphate with a specific man-made reactant and the “reactive” silica being the Silica dissolved in this specific reactant. In fact these man made artificial reactants only approximate to the actual reactional conditions and in fact, from a rigorous point of view, have no validity whatsoever.

In industrial conditions all silica compounds are soluble in HF to some extent, even quartz. As silica is progressively dissolved by the reaction media, the free HF is passivated progressively. Thus the reactant initiates by dissolving the most soluble silica, the diatomites, and whilst free HF is still in existence passes to progressively dissolve the more difficult silica compounds until if there is still free HF even quartz is finally attacked.

This means that although there are subsequently references to industrial cases, which apply perhaps in most cases, there may well be a typical behaviour in specific cases due to variations in the soluble Si to F ratio.

## **2 The Production of Single - superphosphate**

The recovery of fluosilicic acid has been effected for many years. The type of scrubber proposed by TVA was a self-venting eductor/venturi scrubber with three or more stages. The final stage producing acid at 18-20%  $\text{H}_2\text{SiF}_6$ . This equipment is extremely effective in producing a relatively high strength FSA but often the acid contains precipitated silica and the  $\text{P}_2\text{O}_5$  content of the produced acid is above the tolerance for use in the manufacture of  $\text{AlF}_3$ . However the acid is sometimes used for  $\text{Na}_2\text{SiF}_6$  &  $\text{K}_2\text{SiF}_6$  manufacture.

The use of void towers is also proposed in some of the articles and some interesting data on the design of such units is provided.

The recycling of this FSA to the superphosphate den is reported in many of the referenced articles at the end of this presentation but this does lead to an increased load on the scrubbing system. Articles from Montedison and several plants in New Zealand describe these recycling techniques.

## **3 The Production of Triple - superphosphate**

The production of powder TSP in a den is similar to that for the production of SSP. The main difference is that strong phosphoric acid substitutes the sulphuric acid. As can be seen in the following section, on phosphoric acid concentration most of the fluorine in the acid is stripped during concentration. As such, and as the ratio of acid  $\text{P}_2\text{O}_5$  to phosphate  $\text{P}_2\text{O}_5$  is of the order of 2.3 :1 the amount of fluorine evolved is very much smaller than in the case of SSP. This in general means that the condensation of water vapour in the den scrubber is greater than the absorption of fluorine. Thus the production of FSA at a reasonable concentration is normally extremely difficult.

In the granular TSP route, the acid strength used is somewhat lower than in the den route and thus the amount of Fluorine in the feed phosphoric acid is higher. Even so the production of FSA at a reasonable strength is still extremely difficult.

## **4 The Production of Phosphoric Acid**

Fluorine recovery in the gas scrubbing and flash cooler sections of a dihydrate phosphoric acid process is fraught with dangers. The Silica to Fluorine ratio in these conditions is so high that there is almost always a deposition of silica during absorption.

### *4.1 Gas scrubbing section*

In the gas scrubbing sections of all processes, both with Dihydrate or Hemihydrate attack, the FSA

produced must be maintained at a low concentration to prevent deposition of silica. All ducts should be irrigated and in the case of high strength processes in particular, care should be taken to design adequate slopes and irrigation of all ducts.

#### 4.2 Flash-cooling section

The higher the acid strength and the lower level of “reactive” silica in the phosphate the higher the F/Si ratio in the FSA produced. However it is not possible to produce a high strength FSA due to the unfavourable Si/F ratio.

In the dihydrate process PRAYON uses a “pre-condenser” which heats the filter wash water and prevents some of the fluorine from the vapours ex-flash cooler reaching the cooling towers or cooling-pond. Although no aim is made to produce a concentrated stream.

Hydro Agri in Holland operated a single strength hemihydrate plant producing 50% acid and used the HF rich FSA from the evaporation units as make up and analysing the F/Si ratio until the ratio decreasing was 5. At this point the scrubber was drained and refilled with HF rich FSA from the evaporators. This is one of the clearest proofs of the existence of the compound  $\text{H}_2\text{SiF}_6 \cdot \text{SiF}_4$ , as the solution is clear up to the ratio of 5 but below this the solution is milky.

### 5 The Evaporation of Phosphoric Acid

In general there are two flow sheets used for the production of concentrated acid from the ex-filter acid, firstly by single-stage evaporation and secondly by multi-stage evaporation. The F/Si ratio of the evaporated vapour increases with increasing concentration. As a general rule single stage evaporation from ex-filter acid produces a milky, Si rich solution up to 46-48%  $\text{P}_2\text{O}_5$  whereas above this value the solution is clear. Clear means  $\text{F/Si} > 5$  ( $\text{H}_2\text{SiF}_6 \cdot \text{SiF}_4$ ).

This means that if FSA is recovered from a multi-stage system then the make up should be fed to the recovery system on the high strength evaporator and that the HF rich FSA produced from this evaporator is bled to the recovery system on the low strength evaporator. In this way, a clear FSA can be maintained in both systems and all scaling minimised.

One of the first processes developed for the recovery of FSA during the evaporation of phosphoric acid was the “Swift” process. This process was patented and licensed to a large number of companies throughout the world. The main disadvantage of this process was the size of the fluorine scrubber which was about 10% larger in diameter than the evaporator itself. This size was important to minimise the upward vertical velocity of the vapours so that they would not entrain the smaller droplets of the sprayed FSA. This being extremely important as the irrigation rates were many many more times the production rate, thus any entrainment caused a considerable reduction in recovery.

PRAYON has devised a much more compact unit that irrigates a duct with a very small cross-section. thus reducing the volume of FSA irrigating this smaller section. This saves considerable pumping energy and allows much smaller plastic pumps to be used. The system is also co-current which allows the droplets to impinge on a wetted surface reducing entrainment. The gas then reverses direction and passes through a specially designed droplet separator to recover any very small particles still entrained. This unit is very much smaller than the old “Swift” design, it is cheaper to build and also being very compact is useful for squeezing between existing units when revamping existing units

### 6 The Defluorination of Phosphoric Acid

The defluorination of phosphoric acid can be effected by two distinct methods of stripping one by air and one by steam.

In both methods additional Silica is added to complex the Fluorine and aid the stripping process. This means that the ion  $\text{SiF}_6^{2-}$  is formed in the solution and  $\text{SiF}_4$  is stripped from solution. The presence of Aluminium and Magnesium make defluorination more difficult due to the presence of complex ions in the solution. The Aluminium ion is known to be  $\text{AlF}_6^{3-}$  but the complex Magnesium ion is still not confirmed.

In both processes FSA can be scrubbed from the vapours or gases leaving the defluorination vessel but the presence of large quantities makes the utilisation of the FSA produced somewhat difficult.

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