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**FIFTY YEARS OF PHOSPHATE PROCESSING TECHNOLOGY**  
**OR**  
**“THE BONES OF PHOSPHATE TECHNOLOGY”**

**by**

**Paul A. Smith, Société Chimique Prayon-Rupel, Belgium and**  
**John Sinden, JEATECH, Brazil**

# FIFTY YEARS OF PHOSPHATE PROCESSING TECHNOLOGY<sup>1</sup> OR “THE BONES OF PHOSPHATE TECHNOLOGY”

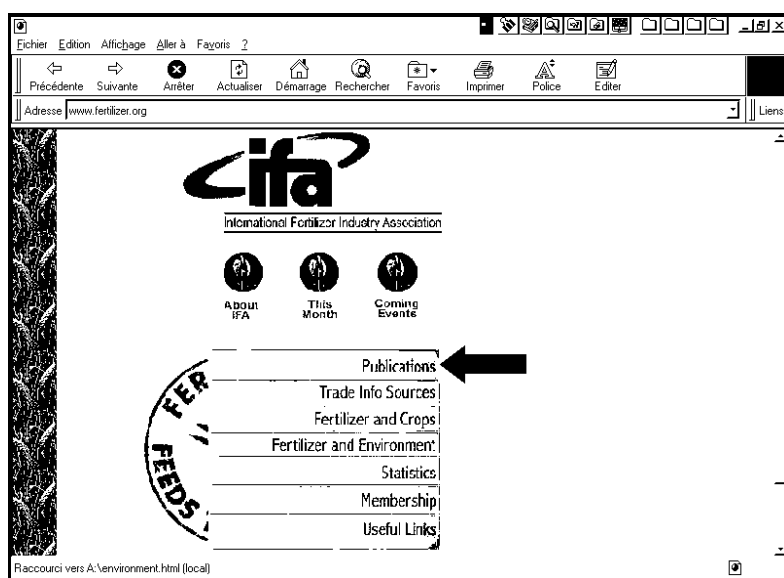
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## Introduction

When the proposal of an Exceptional 50th Anniversary Technical Conference was initially mooted, the idea of a visionary presentation on potential technological developments that could be expected in the future was envisaged. Although this type of analysis may well be applicable to nitrogenous fertiliser technology, the utilization of such an approach in the case of phosphate and granulation technologies must be doubted due to the difference in the basic technological concepts.

This presentation shows without doubt the contribution the ISMA/IFA Technical Conferences have made to the dissemination of technology for the benefit of all.



Many people in the industry still believe that a certain part of phosphate & NPK technology is “artisanal”, this is certainly not true as today’s understanding of the chemical and physical processes is better than ever. This still does, however, not allow the “gurus” of today to develop computer programs that may be used blindly by the future generations. The main basis for a good understanding is a rigorous technical base combined with a detailed knowledge of the practical experience already gained by the industry. Of the young engineers arriving into the industry today although most are conscientious, technically competent & computer literate some of them lack the desire to read books and articles but prefer to “surf the net”.

Now their dreams have come true, the IFA Information Service has put all the papers presented at the ISMA/IFA Technical Conferences into an index and this can be accessed via the IFA Web Page <http://www.fertilizer.org>. Access to the index could be no simpler, on opening <http://www.fertilizer.org> the “Home Page” of IFA appears as shown above, one “click” on “Publications” reveals the publications page (Figure 1) and one more “click” on “IFA Technical Papers” reveals the full Index, see Appendix 1. An *off-line* “key-word” driven search engine exists to

<sup>1</sup> Paper presented at the IFA Technical Committee Meeting, “50<sup>th</sup> Anniversary of the IFA Technical Conference”, 25-26 September 1997, Seville, Spain

assist them to find articles pertinent to specific subjects easily. This service is accessible to member organizations through the sending of a simple *e-mail* containing the relevant “keywords” addressed to the IFA Information Service. Another excellent index is that of the “Proceedings of The Fertiliser Society”<sup>1</sup> that also contains many interesting articles on Phosphate, Phosphoric Acid and NPKs some of which are referred to in this article.

**Figure 1 - Publications Page**

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**Publications**

Extracts from selected IFA publications have been made available on-line:

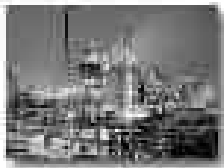
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## Structure

The IFA Information Service has taken a considerable time in creating a data base of all the papers presented at the 25 Technical Conferences already held (the latest issue being “Index of ISMA/IFA Technical Conferences 1947-1996”) and this in itself is a very useful tool for the “apprentice” in the phosphate field. The aim of this presentation is to assist these newcomers by giving an insight into the gems of information contained in these “ancient texts” in order to stimulate the use of this “history” and prevent the reinvention of the wheel. The articles mentioned in the following texts are not intended to be exhaustive but have been selected solely to show how much useful information is available from this source.

The first part of the paper dealing with **Phosphates and Phosphoric Acid** was written by Paul. A. Smith and the format of this Index will be followed as a basis for the structure of this article and a marked up version of the Index with the papers in each section numbered from “a” through to “z” and then “A” through to “Z” is attached as Appendix 1 to aid cross referencing of this paper to the articles.

The second part, dealing with **Superphosphates and Granulation**, was written by John Sinden and will discuss some of the classic papers presented on this subject.

### **A. Phosphates and Phosphoric Acid** by **Paul A. Smith**

#### **1. Phosphates**

##### **1.1 Characterization of phosphates and pilot-plant testing**

Many papers deal with the aspect of phosphate structure and the effect on “reactivity” (See Appendix 1 - 1.2.d, 1.2.e, 1.2.f, 1.2.g & 1.2.x). However, one must always treat the aspect of “reactivity” with some caution. “Reactivity” applies to the potential reaction rate under ideal conditions. This is useful for the reactions in the production of TSP when the effect of particle size is duly considered but the effect of “coating” when the sulphate ion is present makes this data meaningless for reactions related to SSP and phosphoric acid manufacture. The article 2.1.j in the section Phosphoric Acid has an excellent treatise with respect to “coating” of igneous phosphates.

Other papers deal with the subject of pilot-plant testing for phosphoric acid manufacture. The most interesting paper on this subject, actually classified 2.1.z, dealing with the comparative results of a pure phosphate “spiked” with various naturally occurring contaminants. A further paper on this subject was presented at the IFA/CNCCC meeting in Beijing in 1983 (“Selective flotation of apatite from carbonatite glimmerite ore and use of this concentrate for phosphoric acid production at the Siilinjärvi plant and mine of Kemira Oy, Finland”).

Also at this Beijing conference there was an excellent presentation by N. Robinson "Procedures to Evaluate Phosphate Rocks for Phosphoric Acid Manufacture" that complemented a first paper classified as 2.1.p that was presented in Orlando and another presented at the ISMA/ANDA conference in São Paulo in 1975, "Laboratory and Pilot-plant Assessment of Phosphate Rocks for Phosphoric Acid and Ammonium Phosphate Manufacture".

Within this particular section, 1.2.p studies the effect in pilot-plant of higher MgO levels whilst the paper 1.2.r although not totally rigorous does contain some interesting details. Pierre Becker also presented a paper on the pilot-plant study of corrosion which was classified 2.1.t.

## 1.2 Phosphate selection

The selection of a phosphate for phosphoric acid manufacture is a very important decision. The level of beneficiation in a mine site case affects the overall production cost whilst in the case where the client buys his phosphate on the world market, it is important to choose the one which gives him the lowest production cost and a product quality that is acceptable. The first paper, which has some very interesting points, was 1.2.h. by M. H. Hampton of SAI. This was followed by that of the Italians presented in Orlando, 1.2.s and that of Hallsworth & Enriquez 1.2.v. Another paper classified 2.1.f gives Rhône-Poulenc's views on the subject but there is one error as it is the CaO/P<sub>2</sub>O<sub>5</sub> that gives the guide to sulphuric acid consumption after the efficiency has been taken into account. Some of the aims of the beneficiation process to produce an optimum quality are set out in the Jordanian papers of Hummadi et al 1.2.C and Ghoseh et al 1.2.E and also 1.2.F by Guedri & Lahmadi.

## 2. **Phosphoric Acid**

### 2.1 **General processes - dihydrate processes**

#### 2.1.1 Early technology

At the first conference held in Landskrona in 1947, a paper classified 4.2.a deals with SSP and TSP but states that the Belt-filter was invented by Wallny and some sketches are included of the original machine used in phosphoric acid. This article was complemented by a paper presented by Union Chimique Belge & Saint Gobain at the 1949 conference in Milan dealing with the Landskrona belt-filter, mechanical defoaming, agitation and reaction volumes, 1.3 m<sup>3</sup>/t Gafsa/hour). The paper, 2.1.b presented by the Italians in Milan was the first to discuss the insoluble loss and the soluble loss. The insoluble loss was 3% and using three countercurrent drum filters with intermediate repulping a filtration efficiency of 98% was obtained. Also the reduction of corrosion due to higher levels of molybdenum in stainless steel was first noted.

Sven & Ralf Nordengren in 1953 at the Cambridge conference covered the aspects of temperature control, slurry recirculation, mechanical control of foam by agitation in the paper classified 2.1.d but the most important fact was the admission that an "overall" efficiency of a unit, when "accidental losses" were considered, was only of the order of 80-90%. It is still true today that "housekeeping" is still a very important factor in maintaining a high overall efficiency.

At the Aarhus conference in 1955 the pioneer utilization of flash-cooling was mentioned by Beetz & Jadot of Prayon (2.1.e) giving a figure of approximately 1 tonne of water being evaporated per tonne of P<sub>2</sub>O<sub>5</sub>. This paper also gave the other principles of the first generation of the Prayon process and stating that industrially an attack efficiency of 97% and a filtration efficiency of 99% were possible giving an overall process efficiency of 96%.

#### 2.1.2 Insoluble losses

The splitting of the insoluble loss into two parts was also mentioned for the first time by the pair from Prayon in paper 2.1.e. They stated that if washed gypsum (free of water soluble P<sub>2</sub>O<sub>5</sub>) was extracted in a large excess of water part of the "insoluble loss" dissolved, the balance being unreacted phosphate. This method is still used by Prayon to determine the co-crystallised loss. This was a breakthrough as prior to this the conditions to minimize the insoluble loss could not be defined.

Further studies of the split of “insoluble loss” were made by Fröchen & Becker in 2.1.h at Stockholm in 1959 where it was surmised that the more soluble part of the insoluble loss was di-calcium phosphate trapped in the calcium sulphate lattice.

These discoveries of the 1950's showed the key to optimization of the operation of a phosphoric acid plant reactor, it is a shame that here on the verge of the millennium many factories still do not check the split of the insoluble loss. The lattice loss or cocrystallised loss can be determined by various methods. Prayon dissolves the washed gypsum in an excess of water, Hydro dissolve in saline water (gypsum is more soluble in sea water) but most people, especially in the USA use the Citrate Soluble method and call the loss “the CS loss”. The CI loss (Citrate insoluble loss) is effectively the unreacted phosphate.

### 2.1.3 Transition - Dihydrate / Hemihydrate

It was also at the Stockholm conference, in paper 2.1.i, that the famous “Dahlgren curve” relating to the transition between dihydrate and hemihydrate was first published. This curve actually defined that the sulphate level affected the transition but sadly the people that later copied the curve left out this important factor.

The ease of conversion of Dihydrate to Hemihydrate is the basis of the Central-Prayon process (2.2.d, 2.1.c & 7.2.m) a unique process which first precipitates the calcium sulphate as gypsum separates off the product acid and then recrystallizes in hemihydrate to recover the cocrystallised loss. The added advantage is that if put to stack the hemihydrate absorbs the free water and produces a virtually dry, powdery dihydrate for immediate down-stream use as a building material.

The converse, recrystallization from hemihydrate to dihydrate, is not always so easy. Miyamoto of Nissan (1.2.P) stated that the conversion of igneous phosphates containing larger amounts of strontium cause the rehydration to be retarded and as such much larger conversion volumes are required.

Rehydration on the hemihydrate filter can be retarded by using additives as claimed by Fisons in 2.2.j but also mentions that fluosilicate scaling can be a problem in the strong wash section of the hemihydrate filter. Miyamoto also mentions this in 2.2.i.

### 2.1.4 Reactor design - Single tank v. multi-tank reactors

Sheldrick also presented a paper in Stockholm, classified 2.3.1.a, giving a mathematical treatise on the simulation of agitation and the advantages of multi-tanks over single tanks for reaction. This was extended to cover other aspects in the paper “Insoluble Losses in Phosphoric Acid Manufacture” presented by Janikowski, Robinson & Sheldrick to The Fertiliser Society in 19, Proceedings N°. 81<sup>1</sup>. Whilst in Edmonton in 1988 Robinson once again showed in paper 2.2.q that a multi-tank design, in this case in hemihydrate units, has considerable advantages with respect to dissolution when treating coarse phosphate feeds. This was also the basis of a Dorrco design to treat coarse phosphates as presented in 2.1.q where it was considered that the annulus of the basically single tank Dorrco unit could be compartmented to treat coarser phosphates.

The concept of single tank, high recirculation, iso-thermal, iso-sulphate conditions being the best design for crystallization was totally destroyed by the SIAPE presentation 2.1.D in Tunisia where they showed that having a zone within the recirculation loop at a lower temperature *improved* the filterability in the case of the SIAPE reactor on Gafsa phosphate.

The Rhône-Poulenc Diplo process (2.1.F) also showed advantages of using two reactors and splitting the reaction volume into two distinct zones but in this case the filtrates from the filter, the mother liquor and the strong wash, are recycled separately to create a  $P_2O_5$  differential between the reactors. The fears of this type of process are principally in the inability to separate fluctuations on the filter from causing fluctuations in the reaction section.

The conversion of a single-tank unit into a multi-tank design and the resulting improvements is the basis of the paper presented by JPMC, 2.1.G in Amman, 1994.

## **2.2 Anhydrite / hemihydrate / hemi-dihydrate processes**

### **2.2.1 Early technology**

Early attempts to produce phosphoric acid whilst precipitating the calcium sulphate in the hemihydrate or anhydrite mode were fraught with problems (2.2.a, 2.2.b & 2.2.c) particularly due to the lack of attention to the critical state of water balance. In 2.2.c Hakansson claims a success but in paper 2.2.k the reconversion of the plant to dihydrate mode is discussed.

### **2.2.2 Dihydrate-Hemihydrate Process**

The Central-Prayon process simultaneously producing a relatively high-strength (37%  $P_2O_5$ ) acid and a merchant grade gypsum capable for use in plaster or cement / sulphuric acid production directly from the filter is covered in 2.2.d. The technological means to produce a high quality by-product gypsum being proved by this process. The sole problem being the relative size of the production of the gypsum and the size of the market for the off-take of such a product.

### **2.2.3 Hemihydrate & Hemidihydrate processes**

The production of high strength acid in a single tank was studied by Rhône-Poulenc in 2.1.g but it was the article 2.2.e presented in Brussels in 1968 which is a classic in the control of crystallization producing a highly filterable hemihydrate and a strong (50%  $P_2O_5$ ) acid. This was followed by the paper 2.2.f presented in Seville in 1972 which gave some light on the industrial results. The disadvantage of this single-stage process is the high co-crystallised  $P_2O_5$  in the hemihydrate rejected and the sensitivity to accidental losses from the filter. Thus the Hemi-dihydrate process was rapidly developed and details of this process were first outlined in 2.2.g presented in Prague in 1974, followed by industrial experience presented by Lasovic et. al. in The Hague 1976 (2.2.j). In this paper along with that by Miyamoto, 2.2.I the problem of fluosilicate scaling in the down legs from the strong-wash section of the filter was highlighted. In the paper 2.1.C the extension of the Central Prayon process with the addition of a hemihydrate stage to produce strong acid is mentioned.

## **2.3 Other topics**

### **2.3.1 Filtration**

Although filtration is specifically referenced in only five papers, there are, in fact, many references in other sections. The paper 2.3.2.c compares the Giorgini travelling pan filter the Prayon filter and the UCEGO filter. The Giorgini method has been replaced by newer designs of belt filters and the Bird-

Prayon and UCEGO of today do not bear much resemblance to those of 1965. The modernization of a filter and the results on the overall operation of the reaction section is covered in the paper 2.3.2.e whilst paper 2.3.2.d covers the mathematical modelling of filtration and washing of gypsum cake. The paper presented by SIAPE on the use of flocculents is also of great interest, 2.1.m.

### 2.3.2 Concentration

Although the numbers have been scrambled so as not to give away the actual design figures the paper of Moraillon and Roubinet, 2.3.3.b covers many important aspects of the design of concentration units. The effect of tube velocity is covered, the need for the full de-superheating of the steam and the care that should be taken with the design of the condensate system are fully covered. The boosting of a unit in Florida is covered in the paper 2.3.3.e and the paper presented in Edmonton, 2.1.E deals with a new alloy used for the circulation pump. Submerged combustion is also covered, 2.3.3.c, in fact one of the few units still operating today is used for liquid fertiliser production at the Engis plant of Prayon.

### 2.3.3 Materials of Construction

I have never ceased to be amazed by the lack of papers on materials of construction. Operating companies would not really be giving away proprietary information and equipment and stainless steel suppliers should be keen to divulge their strengths but even so in fact only three papers are covered in this section 2.3.4. In 1949 Montecatini already noted that the addition of molybdenum is very favourable to the reduction of corrosion and in 2.3.4.a this is restated but including the fact that low carbon steels are more resistant to the wet process acid environment. The paper 2.3.4.b mentions the advantages of HV9A and Uranus B6 due to the higher molybdenum contents compared with the standard Alloy 20. Once again repeating the positive effect of low carbon and high molybdenum.

### 2.3.4 Effluents and Fluorine

Here I have grouped together the sections 2.6, 4.4 and 7.1. The most classical paper, 4.4.a, being that of Whynes and Dee presented in Cambridge in 1963. This paper already confirms the presence of the complex  $\text{H}_2\text{SiF}_6 \bullet \text{SiF}_4$  and has some wonderful photos of drops coated with silica. The mole ratio of F:Si defines if the FSA produced will be perfectly clear or will have some solid silica in suspension. To have a clear solution the ratio must be greater than 5.0 and not 6.0 as might be suspected ( $\text{H}_2\text{SiF}_6$ ) due to the formation of the compound  $\text{H}_2\text{SiF}_6 \bullet \text{SiF}_4$ . This is also mentioned in the Paper, 9.3.j, presented by Windmill in The Hague in 1976. Many papers deal with the evolution of fluorine from SSP/TSP units whilst the paper 2.6.g, presented by the Italians, mentions how FSA can be recycled to a phosphoric acid attack system. The paper 2.6.h deals with the F/SiO<sub>2</sub> ratio and its effect on the evolution of fluorine but one of the main modifiers to the fluorine balance is Al<sub>2</sub>O<sub>3</sub> which forms the ion  $\text{AlF}_6^{3-}$  which holds the fluorine in the acid up to the concentration stage. Magnesium also has a similar effect but the actual ion formed has not been identified. Both Al and Mg linking with the fluorine make the acid less corrosive. The classic paper on fluorine recovery from evaporation units is that presented by Swift in 1963, nowadays more compact units to recover fluorine are being designed.

### 2.3.5 Gypsum

Many papers have dealt with the “gypsum problem”, there is in fact no problem at all in producing a merchant grade gypsum and phosphoric acid at the same time, the paper presented in 1988 at



Edmonton proves this, the only problem is that the market for this “co-product” is normally not sufficient to consume all the production. The use of gypsum to make sulphuric acid and cement was first mooted in 1949 but in fact the rules of physical chemistry have not changed over the years and this process still requires an enormous amount of energy to breakdown the gypsum.

### **3. Summary**

I must apologise if I have forgotten one or two of the important papers but the aim was solely to whet the appetite and show what gems do exist. I hope that now some of the younger engineers will be convinced that to spend a few hours reading up on the past, with the aid of the IFA Information Service, will be minutes very well spent. Well that concludes the part on "Phosphates and Phosphoric Acid", this paper will now be followed by John Sinden's contribution on "Superphosphates & Granulation".

## **B. Superphosphates and Granulation**

by  
**John Sinden**

### **1. Introduction**

When first asked to co-author this paper I agreed that a paper such as this could be a stimulus to the younger members of the industry by showing how much useful information is already available in both the IFA Information Service and the "Proceedings of the Fertiliser Society", but it also brought back some vivid memories.

The first one, very early on in my career, concerned one of my managers. He said "John if you have a good filing system and a good memory very shortly people will start to take notice of how easily you solve the problems. This is because there really is very little that is new under the sun and the problems come round in circles. So if you know what happened last time it is easy". He also said that solving the problem was only half of the work the second half was reporting it to the right people.

The second memory is more recent, when I was Technical Manager for a major Brazilian fertilizer producer, when I became involved in a long discussion with the company's President about what was the most economical conversion of the phosphate rock when producing single superphosphate. He was insisting on trying to achieve the highest possible conversion. The data which we were generating in the production control department showed that the highest conversion was not the most economical.

After several months of collecting more data and doing a large number of extra calculations it was finally possible to convince the President that he was wrong. By a strange coincidence several months latter I came across the paper 4.1.a which had been presented by P. Cambau at Landskrona in 1947. "Contribution to the Research of the Optimum Acidulation in the Manufacture of Superphosphates". To me for this personal reason this was a very important paper. It was one of the first papers to stress the economical aspects of the industry. It also must be one of the earliest examples of the now classic "J" curve of economics. It is interesting to note that this original work had been initiated by an apparent inconsistency. That was a that a SSP had been produced and sold with 1.5% insoluble  $P_2O_5$  was more profitable than the SSP made with only 0.5% insoluble  $P_2O_5$ .

## **2. Phosphates and Superphosphates**

### **2.1 Fundamental Concepts and Design Data**

The concept of "Reactivity" as stated in section 2.1 is very different for the production of TSP and there is just as wide a difference between the effect of the sulfuric acid in the production of SSP and phosphoric acid. Of the papers which treat this subject (1.2.c, 1.2.d, 1.2.i and 1.2.B). The most important is 1.2.d and its complementary paper of the same title given as Proceeding n° 42 of the Fertilizer Society<sup>1</sup>. This treats the rock composition and source - sedimentary or igneous as the most critical factors.

Because of the large difference in construction costs of the superphosphate units and a phosphoric acid plants, it is normal for companies to build a superphosphate unit without carrying out extensive or any pilot plant tests. This is not the case for phosphoric acid units. These can be clearly seen from the titles in section 2.1 where there has been many papers on this subject.

### **2.2 Early designs of superphosphate units**

In the first 14 years there were 17 papers on the manufacture of superphosphates as compared to 3 in the last 21 years. It is easy to see why ISMA became IFA. Of these papers; we have already discussed 4.1.a. The other papers which had significant effects and are still useful today were the papers 4.2.c, 4.2.c and 4.2.j which described the three main production processes. Two using easy to construct belt dens and the other describing the modifications to the more traditional slat type den. Today the simplest, the Etablissements Kuhlman linear belt den, having the lowest investment and maintenance costs has dominated the market in the countries which still make significant production of superphosphates - Brazil, Egypt, India, Australia, New Zealand and even the large plants in China.

The other processes such as were described in papers 4.2.i and 4.2.q have basically fallen by the wayside.

It is interesting to note that for such a significantly important commercial process the Kuhlman paper 4.2.d is very brief and tries not to give away practical details.

In this section there were papers (4.2.c and 4.2.p) devoted to controlling the production and how to calculate your production. This still remains a problem today, with a product which undergoes "curing" in the bulk product store - losing moisture and other gaseous components both of which are variable - as a more weighty product is of little use.

Papers (4.2.h) which was a work by the authors of paper (1.2.d) studied the process variables in the production of SSP in more detail. The paper also cited the problems of being able to analyze the products where the preparation methods used in wet chemical analysis seriously changes the chemical composition. The primary difficulties being with moisture content and free acidity. It is to be noted that the problems do not go away. The paper (11.t) was about the problems of the determination of moisture contents.

There have been very few papers about TSP, of these none were particularly significant, the first being a brief paper describing the possibilities of making the product economically (4.2.f) the other on how it was necessary for certain types of phosphates to use surface active agents to improve the physical properties and granulation characteristics (papers 4.2.n, 4.2.r and 4.2.t).

There was no paper comparable with the Fertilizer Society Proceedings n°. 21<sup>1</sup> "The Manufacture of Triple Superphosphate" by J.J. Porter and J. Frisken in 1953, which went into great detail on the subject.

### 2.3 Ammoniation of Superphosphate and Fluorine effluents

The remaining papers in section 4.3 and 4.4 deal with the granulation and ammoniation of the superphosphates and with fluorine effluents. Of these papers only (4.3.d) on the ammoniation and granulation could be considered significant, but since this work was carried out in UK which had at that time guarantees for phosphorus based on water solubility the degree of ammoniation was strictly limited.

Fluorine effluents have always been a part of the superphosphate story as the principal environmental concern. The papers, 4.4.a, 4.4.b, 4.4.c and 4.4.f, treated the chemistry of the system. The most important paper was 4.4.i which treated the recycling of the fluorine effluent by-product, the  $H_2SiF_6$ , back to the reaction system and to extinction. This was demonstrated by the Italians in this paper, which in the case of sedimentary phosphate rocks had the following advantages, reduction in the sulfuric acid consumption and the disposal of the by-product  $H_2SiF_6$ .

## 3. **Granulation**

It is going to be very difficult to limit this review to papers that only considered the granulation of phosphates so we have included some of the NP and NPK fertilizer granulation papers.

### 3.1 General

The following papers (5.1.f, 5.1.l, 5.1.m and 5.1.o) all made significant contributions to taking away some of the "black art" from the granulation processes. T.P. Hignett's paper described the various developments that had been made at the NFDC - TVA.

The 1976 meeting at The Hague was particularly rich in granulation papers (5.1.j, 5.1.k and 5.1.m) all being important in their different ways - 5.1.j being on a specific process route which used a mixture of potassium chloride, phosphate rock and sulfuric and phosphoric acid to make granular superphosphates and PK compounds in a single step process. The control of the system becomes very critical particularly in the drying section to avoid the formation of excessive amounts of hydrochloric acid gas.

The paper 5.1.l was for me a follow up paper by CROS S.A. on the applications of their pipe reactor. This time being used with urea to make high analysis - ceiling grade products such as 1:1:1 (19-19-19) at low cost and at relatively low recycle rates. This was directed at the upgrading of the conventional regional type granulation plants based on solid raw materials.

The paper 5.1.m on the other hand described the advances carried out by Jacobs Engineering to the traditional high recycle slurry based routes to NP and NPK products. These plants were originally built in the phosphate region of central Florida but are also used in many other parts of the world. The new details included controlling the recycle and also improvements in the crushing of the oversize and obtaining an equilibrium between the dryer and granulator.

A common feature to all these papers was that they were all describing specific process routes and did little to explain the theory or mechanisms which lead to the granulation of the raw materials.

### 3.2 Ammonium Phosphates - Section 5.2 of the Index

Again the papers in section 5.2 "Ammoniation, MAP and DAP" are mainly descriptive of specific processes or process routes such as papers 5.2.d, 5.2.e, 5.2.g, 5.2.h, 5.2.i, 5.2.m, 5.2.n and 5.2.r or they concentrate on the effects of the impurities in the phosphoric acid in the product quality and/or production capacity - these were papers 5.2.o, 5.2.q and 5.2.s. There were fewer papers being directed towards the chemistry of the systems but these were more concerned about product quality, chemical and physical, rather than the actual processes of making good granules. In this category were papers 5.2.b and 5.2.j.

There was a general tendency for these process based papers to be related to product cycles. One of these cycles was the use of powder MAP as a substitute for superphosphates as a way of permitting the smaller regional granulation plants which had been designed to use basically solid raw materials to make higher analysis products without having to invest in storage capacities for the fertiliser liquids - ammonia - sulfuric and phosphoric acids.

Hence there are various papers discussing the relative merits of the different routes used to make powder MAP. During the late 1960's through to the early 1980's more than 40 of these type of plants were built in Europe, North America, North Africa, The Middle East and in many other parts of the world. Very few are still operating today, with even fewer number being built. The main use for these units today is that with the decline in the production of TSP they are one of the only routes capable of consuming the "sludge" from the clarification and purification stages of phosphoric acid production.

One of the more interesting papers in this section was presented in Paris (5.2.s). This was a pilot plant scale investigation into the effects of the impurities in the phosphoric acid on the production of DAP. The main impurities studied were iron, magnesium, aluminium and fluorine. It is interesting to note that the North American, and more specifically the Florida based companies, have a phobia about high levels of magnesium and their effects on both the production of the phosphoric acid and the DAP. One suspects that this study was carried out to confirm their suspicions about magnesium.

The conclusions of these tests were as follows:

- I. Aluminium showed the most severe effect on operations. It also affected the DAP grade by decreasing to the ability to incorporate nitrogen thus decreasing the  $\text{NH}_3 : \text{H}_3\text{PO}_4$  mole ratio. It caused more problems with viscosity in the preneutralizer, but improved the storage properties at high moisture levels.
- II. Iron showed the most effect on nitrogen grade, but showed less effect than aluminium on a weight for weight basis on plant operation. It also adversely effect preneutralizer viscosities but not as much as aluminium. It caused some changes in the physical properties of the granular product.
- III. Magnesium also lowered nitrogen grade but not as much as iron. The magnesium reduced the formation of citrate insoluble  $\text{P}_2\text{O}_5$  and improved the neutralizer slurry viscosity! There were some negative effects on the products storage properties.

Section 5.3 deals with nitrophosphates which are outside the scope of this paper.

### 3.3 Special Techniques - Section 5.4 of the Index

In the section 5.4 of the Index some of the most important papers are classified. In the first section 5.4.1 - Reactors, each paper is once again more descriptive of specific process routes and equipment the most interesting papers being (5.4.1.a, 5.4.1.b, 5.4.1.d and 5.4.1.e). With paper (5.4.1.b) given by B.R. Parker of TVA at Orlando in 1978, details not only of process parameters but also some design parameters are given, but once again specifically for the equipment and not the mechanism of the granulation process.

In section 5.4.2. Granulation, there are several papers which helped demystify the granulation process. These include papers (5.4.2.d, 5.4.2.g, 5.4.2.m) while other papers on this aspect can be found in (9.2.h, 10.g and 10.k).

Besides these papers there were also the Fertiliser Society Proceedings<sup>1</sup> N°. 47, 55, 59, 127, 215 and 310 - and another excellent paper was given by J.A. Benes at the IFA Middle East Regional Technical Seminar on NPK Fertiliser Technology in Aqaba in 1983. Of all these papers the one which is of special interest and importance is 5.4.2.e "Agglomerate Granulation as an Equilibrium Process" presented at the IFA Technical Conference in The Hague in 1976 by J.H. van der Leek. This paper not only treats and explains the relationship between the ratio of solid and liquid phases in the granulator but also treats the variations which can occur in drying process. It also explains effects that the efficiencies of the screens and the oversize crushers have on the process of granulation. It is obvious from the way J. Benes talked about van der Leek's paper in his various presentations that it had a major influence on the GPSM - Granulation Process Simulation Model that was developed at by UHDE G.m.b.h. and presented at various technical conferences. The first being "Design of Compound Fertiliser Plants with the Aid of Computer Models" IFA Regional Meeting, Aqaba 1983 and the latest paper 9.2.h "Modifying Existing DAP/NPK Plants to Comply with New Environmental Regulations", Amman 1994. Basically the UHDE Benes' papers simplified the J.H. van der Leek's conception and theory on the agglomeration process. They introduced a "granulation factor -  $\alpha$ " which was very easy to measure and this plus various series of screen analyses made it possible to evaluate the operation of a granulation plant and build a simple but accurate model of the granulation plant and process.

One of the earliest classic papers was given by at the Fertiliser Society by A.T. Brooks "Developments in Granulation Techniques" in 1957 N°. 47. The basic change here was the use of steam instead of water as the granulation medium.

It went in to describe the effects of the particle size distribution of the raw material - the recycling average the distribution of the liquid phase and the rotation of the granulation drum.

The final paper would have to be paper (10.k) "Operating Experience with Phase Rate Based Control of the CAN/NPK Granulation" by P.B. Olsen of Kemira which was presented at The Hague in 1992. This is the view to the future using an expert system integrated to a Phase Rule Control System which allows the use of a DCS system to control the granulation process using fuzzy logic.

The system is supposed to be user friendly and as good as the best human operator.

#### 4. Conclusions

Before writing this paper both Paul Smith and myself were convinced that the task would not be easy and that such a paper would be “very dry”. We both have tried to demonstrate, each in our own field, that extensive useful information is available in the annals of the IFA Information Service and that via the advances in information technology tracking down such information is easier than one might expect. I’m sure that we have both left out significant presentations but at least all people reading this document are aware of the depth of information available and may be stimulated to read at least one or two of the articles.

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# **Appendix 1**

## **Index of ISMA/IFA**

### **Technical Conferences**

**1947 - 1996**

Claudine Putz

IFA Information Service



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