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THE FILTRATION OF PHOSPHORIC ACID LIQUOR:

DIFFERENT TYPES OF FILTERS TO DEAL WITH

THE INCREASED CONCENTRATION OF ACIDS PRODUCED

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The concentration of wet-process phosphoric acid was about 20% P2O5 at the time when the first installations were constructed and has continually increased ever since. This increase in concentration is essentially due firstly to progress achieved in phosphate rock reaction methods and secondly to filter improvements. At present, concentrations of 30-32% P2O5 are normal for certain processes, and it will soon be not unusual to do considerably better.

In previous papers it was shown that a single reactor tank without internal partitioning (1), apart from its robustness and its simplicity of construction and installation, offered better possibilities for preparing phosphoric slurries of a high P2O5 concentration (2).

We do not wish to revert to this question of preparing phosphoric slurries but to deal in this discussion with a particular related subject. We wish to restrict ourselves to the study of different types of filters in relation to their use in the production of an acid of high concentration. We shall therefore deliberately ignore certain factors governing good filtration which are not related to filters. We shall try to describe the main problems and difficulties which filters must resolve and bring out their importance when the concentration of the acid produced increases.

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- (1) Patent taken jointly by Saint-Gobain and Union Chimique Belge.
- (2a) Cf paper by Saint-Gobain: "The Possibility of Making 45% P2O5 Phosphoric Acid by the Gypsum Process" - ISMA Technical Meetings, 1957, Ref. LE/904.
- (2b) Cf paper by S.E. Dahlgren: "Physico-Chemical Background of Phosphoric Acid Manufacture by Wet Processes" - ISMA Technical Meetings, 1959, Ref. LE/59/61.

Finally, within this context, we should like to put forward certain ideas and observations from our experience and experimentation, together with certain conclusions on the more usual types of filters, in order, on the one hand, to clarify certain points as they appear to the user and, on the other hand, to draw the attention of filter constructors to certain requirements at a time when acid producers are becoming more and more demanding with regard to filter performance and possibilities.

I. THE FILTERS

Different types of vacuum filters are in present use, each type (or very similar types) being put forward by different constructors. We shall mention:

I.1 Rotating Filters

1.11 Turning table filters (with a continuous surface of cake) e.g.: Dorr filters - Imperial filters.

1.12 Tilting pan filters, e.g.: Eimco filters - Prayon filters.

I.2 Rectilinear Filters

1.21 Travelling belt filters (with continuous surface of cake) e.g.: Lurgi filters - Philippe filters, etc..

1.22 Tray belt filters, e.g.: Giorgini filters - Nordengren filters, etc..

1.23 We shall also say a few words about drum filters (with continuous surface of cake) although their use can not be considered without intermediate re-pulping stages.

2. THE CLASSICAL PROBLEMS RELATING TO FILTERS AND PHOSPHORIC ACID IN THE CASE OF ACID OF HIGH CONCENTRATION

2.1 Filtration speed (for a given slurry and at production capacity).

This point is always important, for the choice of filtration surface area, i.e. part of the investment, depends upon it. In the case with which we are concerned, this importance is increased because the filtration characteristics of gypsum decrease in proportion to an increasing concentration of acid produced. This is caused by the viscosities of the acid and the wash solution, both of which increase with an increase in the concentration of acid produced.

Any filter capable of increasing production per unit of filtration surface becomes of still greater interest for a highly concentrated acid than for a normal acid.

2.2 Exhaustion of the Filter-Cake: The problem here is rendered more delicate because:

2.21 The filtration characteristics of the gypsum are less favourable.

2.22 The acid permeating the filter-cake is of higher concentration.

2,23 The water ratio is less favourable. In this regard it is worth noting incidentally that sulphuric acid of the highest possible concentration should be used and that, if for any reason, it is impossible to use concentrated sulphuric acid, this would constitute a considerable handicap for the production of highly concentrated phosphoric acid.

It is also worth recalling, again in connection with the water ratio, that the two classic means of cooling the slurry, viz. evaporation under vacuum and evaporation at atmospheric pressure by the action of a current of air, of part of the water contained in the slurry, are almost equivalent.

2,24 The effect of an imperfect separation of the filtrates corresponding to the different washings will be much larger. Strict attention should be paid to this aspect of filter working, because the quantities of liquid available for washing are smaller and all the concentrations are higher.

2,3 Dilution of the Acid produced during Filtration

This represents almost a special case of bad separation of the filtrates. With the classic filters, a more or less considerable dilution of the acid occurs at the filter face, because the water which penetrates the filter and the filter cloth during washing of the latter is not completely evacuated. This point becomes of very great importance with the increase in the concentration of acid produced.

It can easily be seen that the loss of concentration by dilution from the filter face varies approximately in proportion to the square of the concentration of the acid produced. Thus a filter which would dilute a normally 20% acid to 19% P2O5 would dilute a normally 40% acid to 35% P2O5, all other things being equal (filtration speed, flow of P2O5 in t/hr etc.).

In practice this dilution will be still larger, for the production capacity of the filter will be considerably reduced with increased acid concentration, whilst the amount of water or dilution liquid will remain the same. A filter which, when producing relatively weak acid, is acceptable from the point of view of dilution, very quickly becomes unacceptable when producing a concentrated acid.

2,4 Corrosion and Maintenance

Corrosion of the part in contact with the concentrated acid becomes accentuated, and certain materials and methods of assembly are no longer suitable.

3. THE CLASSICAL SOLUTIONS TO THE PRECEDING PROBLEMS ADOPTED ON THE MAIN TYPES OF VACUUM FILTERS

3,1 Filtration Speed and Production Capacity

As is well known, it is thought to increase filtration speed, and hence production, by means of a high vacuum on the one hand and a small filter-cake thickness on the other.

We have carried out a number of trials and measurements on this subject and, with regard to the subject with which we are

now concerned, filters, we shall only say that they afforded us results which were slightly different but, in general, fairly close to Poiseuille's Law, the gain to be expected from a decrease in filter-cake density and particularly from an increase in the vacuum being slightly less than is indicated by the law.

3.11 Vacuum: The main factor contributing to a good vacuum is the suction boxes. In general, the circular suction boxes of the rotating filters (drum filters, or tilting pan filters of the Binco or Prayon type or turning table filters of the Dorr type, for example), by virtue of their compactness, are those which most easily enable a good vacuum to be attained. It is more difficult to attain good results from the rectilinear suction boxes of the belt or tray-belt filters, particularly after they have been used for a certain time. This is because of their length and also because the rubber belts are relatively more flexible and wear out more easily. We shall, indeed, see later that this lack of compactness has certain advantages.

3.12 Thickness of Filter Cake: The thickness of the cake varies proportionately with the filtration surface area which spreads under the slurry distributor. This filtration surface is in turn proportional to the product of the useful width of the filter and its speed. Thus a wide filter advancing slowly and a narrow filter advancing quickly could be equivalent. It would therefore be wrong to say a priori when comparing several filters, that their production would be proportional to their surfaces. This would only be true if these filters operate at equivalent speeds (speeds at which the same thickness of cake is obtained for rates of flow of the same slurry proportional to the useful surface of the filters).

Geometrically homothetic filters should have homothetic speeds in order to have rates of production proportional to their useful surface areas. In reality, this is not the case and large filters always have lower specific rates of production than small filters, for not only are their speeds not in homothetic relationship, but, for mechanical reasons, they are, in absolute terms, generally not even equal to those obtainable with small filters. We shall indeed see that there exist still further causes of limitation of speed which are also particularly operative for large filters.

For belt filters or tray belt filters, from the mechanical point of view, the speed limitation is above all caused by the resistance of the rubber belt.

The speed limitation for rotating filters of the tilting pan type stems mostly, from the mechanical point of view, from the turnover mechanism for the pans. The friction of the rollers tilting the pans increases considerably with an increase in their speed, which is, in turn, proportional to the speed of rotation of the filter and to its diameter.

The influence of the size of the filter, or, to a lesser degree, of its compactness thus becomes apparent:

at equal peripheral speeds a small filter, or a compact filter, will have a greater production per unit of useful surface area.

Drum or turning table filters have no such mechanical limitations.

It should also be noted that the filtration surface of the largest belt or tray belt filters in existence at the present time is fairly considerably lower than that for the largest rotating tilting pan filters.

3.2 Exhaustion of the Filter Cake

We have seen the complexity of this problem for a concentrated acid. The results to be expected from a filter will depend on certain features of its construction.

3.21 Number of Washings

The greater the number of washings, the easier it becomes to exhaust the filter cake. If we examine the various types of filters, we note that:

Drum filters are very badly placed from this point of view, because only a small wash can, in practice, be made. To improve this situation, one would have to repulp and have additional drum filters.

Rotating filters, either of the "turning table" type (Dorr) or the tilting pan type (Eimco, Prayon), despite the compactness of their suction boxes generally allow three washings. (It would be physically difficult to have more, because of this compactness).

Filters having a rectangular suction box are generally built only for two washings, but can very easily be equipped for more.

3.22 Separation of Filtrates

It is well known that one can have a mixture of two filtrates corresponding to two successive washings either in a container situated under a filter cloth, as the result of too slow a flow of the wash liquors, or when the filtrates arrive in the suction box as the result of a bad fitting of the separations between the compartments of the box.

In this respect, filters having a rectangular suction box have a distinct advantage, firstly because the achievement of a correct separation in the box is much easier because of its length, and secondly because the volume of mixed liquors in the containers under the filter cloth is smaller, either because a considerable slope can be easily achieved, or because a large number of containers can be fitted, and, in any case, because the exit of the liquors occupies a more central position.

However, among the filters with rotating suction boxes, we should distinguish between the tilting pan filters and the turning table filters. For the latter, it is indeed easily possible, if desired, to have the bases of the containers situated under the filter cloth on a considerable slope, because with these filters, one is not

concerned with the question of turning over the trays.

Filter size is also a factor in filtrate separation. The larger a filter, the greater will be the amount of two successive filtrates which become mixed. The passage to be traversed by the wash liquors (and therefore the time necessary for this passage) is never the same for different fractions of a wash liquor spread over the cake surface, certain fractions being directly above the container outlet, whilst others are distant from it. With a large filter these differences are increased. Also, the larger the filter is, the more difficult it becomes to give a slope to the bases of the container under the filter cloth and thus to achieve a rapid evacuation of the liquors they contain. Because of all this, if a good separation of the filtrates is desired (and this is increasingly necessary as the concentration of the acid produced becomes higher) the larger the filter available, the slower must be its speed of rotation. This limitation very quickly becomes more severe than the mechanical limitation and, like the latter, limits the specific production of a filter.

3.23 Possibilities for Regulators

By this, we mean the possibility of easily regulating the position of the wash liquor inlets on the one hand and the suction box separators on the other.

If, with regard to the former, the rotating filters with a vortical axis have a slight advantage, as far as the latter are concerned the rectilinear filters are very much better suited to adjustments in proportion to rates of working, running conditions and conditions relating to the phosphate rock used. Such an adjustment can be made very quickly. Certain distributor boxes on rotating filters can not be so adjusted at all, and this is a regrettable disadvantage.

3.24 Effects of the Pan Partitioning

We have observed from experience that if more or less distinct preferential passages are formed in the cake, it always becomes slightly detached from the partitioning wall of the trays or pans. Although this is not important when large amounts of wash water are available, this phenomenon becomes very prejudicial to a good wash when the volume of available wash liquids decreases. With all filters having a continuous cake surface, this disadvantage is greatly decreased by the absence of such tray or pan partitioning.

3.25 Uniformity of Filtration Conditions

Different filter cake thicknesses, unequal distribution of the wash liquors, and a badly adjusted height of fall of these liquors all lead to irregularities in the washing which become increasingly important as the cake is less thick. It can be readily appreciated that filters with a continuous cake surface are more favourable to the achievement of regular filtration conditions, since with these filters the different continuous solutions comprising the separation between the pans are done away with.

3.26 Filter Cloth Supports

Commonsense indicates, and experience confirms, that it is worth ensuring that the filter cloth support is such that the

holes of this support are distributed in the best possible manner and have the largest possible total area. Filters having a filter cloth support consisting of a piece of rubber are less well placed in this respect than those using a perforated sheet of stainless steel (or a stainless steel grid).

3.27 Washing the Filter Cloths

It is most important to clean off all the gypsum of a previous operation and to wash the filter cloths thoroughly to avoid their becoming encrusted. If the gypsum is not entirely cleaned off, it decreases the free surface of the filter cloth and, by virtue of the water with which it is impregnated, it leads to a dilution of the acid produced. An increase in the resistance of the filter cloths to the passage of the liquids is the consequence of a bad washing. In this regard, turning table filters have a serious disadvantage.

Certain rectilinear filters, on the other hand, have great possibilities in this respect.

3.28 Filter Cloths

We have studied this subject both in the factory and in the laboratory, for if the choice of a filter cloth has an influence on filtration of the phosphoric acid slurry, it also has most important consequences for the manufacture of certain products using the acid produced. (*)

It is generally admitted that single filament cloths are preferable to multi-filament cloths, because the latter become more quickly choked than the former. One more debatable point remains: filter cloth porosity.

We have observed that filtration speed increases at first when filter cloth porosity is increased, but that after a certain point this increase in filtration speed becomes very slight whilst, on the other hand, the amount of impurities passing through the cloth begins to increase very rapidly. It is difficult to clarify this situation more precisely, for it is related to the nature of the slurry and to filtration conditions (or to the type of filter). We may just note that if a filter works with a thick filter cake, the influence of the resistance of the cloth will be relatively less, and in order to have a cleaner acid, one could choose a cloth with a finer mesh; whilst, on the other hand, if a filter can work quickly, the effect of the resistance to the cloth will be greater.

(*) We refer, in particular, to the manufacture of sodium salts (especially the Tripolyphosphate) from wet process phosphoric acid, and to the production of certain granular complex fertilisers. We experienced certain trouble in these processes through having had to use a more impure phosphoric acid than we expected to find.

3.3 Dilution of the Acid Produced

No filter, even a filter which would let fall all its dry gypsum and the cloths of which were not washed with water before each new cycle, is exempt from this risk, and, in any case, the cloths must always be washed.

Different methods are used for evacuating, before filtration of the acid constituting the production, the water or part of the water likely to produce this dilution. These methods may be adapted more or less easily to all types of filter.

However, the same factors making satisfactory separation of the filtrates more difficult on rotating filters and on large filters also make dilution more easy on these filters.

Ultimately, results are the only criterion of efficiency, and it seems that, on certain rotating filters, a fall-off of 1 point in acid concentration is very easily obtained, and sometimes more, for a 30% P2O5 content.

It can be said that at least certain rectilinear filters give distinctly better results (lower than 0.25% decrease in concentration).

3.4 Corrosion and Maintenance

This is too vast a question to be studied in detail. However, it can be said that rotating filters may be considered as robust and as necessitating relatively little maintenance, as long as care has been taken to look after protection against projections. Tray belt filters are more delicate. Rubber belt filters are certainly mechanically the most simple, but periodically the belt tension must be changed, and the cost of this operation is high.

Among the filters actually available on the market at the present time, it is probable that the rubber belt filters would best resist high acid concentration. The construction of the other filters necessitates the use of a considerable amount of stainless steel or other varieties of steel in general use, whilst certain methods of welding in current use could lead to trouble when dealing with highly concentrated phosphoric acid.

5 CONCLUSIONS

Bearing in mind that it is with regard to the production of highly concentrated phosphoric acid that we are seeking the essential qualities of a good filter, we can outline certain general tendencies:

5.1 The desire to make a good extraction on the one hand, and mechanical considerations on the other, lead to a restriction of filtration speeds, which thus limits the production. We seek to ascertain those filters for which this limitation is least serious. A filter which can work only slowly (filters with a thick cake) is less productive and will have a working range limited to low and medium concentrations.

5.2 All things being equal, filters with a small surface area produce the best filtration results; and we may therefore conclude that, very often, despite a larger investment, it would be worth while to install several small filters, which would also have the advantage of giving more flexibility to the plant.

5.3 Although rotating filters are more robust, the effectiveness of their washing operations is less than that for rectilinear filters under similar conditions (No. of washings, water available for this purpose, etc.) because of better separation of the filtrates.

5.4 Filters with a continuous cake surface present least difficulties if the best possible extraction from the cake is desired.

5.5 Many of the remarks which have been made concern filters which actually resolve fairly well the problems arising from the filtration of acid of low or medium concentrations. It is desirable that constructors should improve certain of the solutions they have adopted in order to permit the use of their equipment under good conditions for the production of highly concentrated acid.

These few notes are the results of numerous trials and experience acquired in the use of various types of filters by our company. We hope that many with personal experience of these problems will be stimulated by these fairly general reflections and that they will fill in certain gaps in the picture or correct certain points, for which we thank them in advance.