SOME ASPECTS OF FLOCCULANT USE IN THE PHOSPHATATION PROCESS

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Abstract

The influence on impurity removal exerted by the application of flocculants in the phosflotation process has been investigated. A number of cationic flocculants are shown to enhance the removal of colour and starch. The use of flocculants did not influence the turbidity and conductivity ash of the clarified liquor. Changes in filterability and phosphate content of clear liquor resulting from the application of flocculants are also detailed.

Introduction

Phosflotation (Phosphatation-flotation) is a widely practised method for the defecation of a raw sugar melt. The process involves the coagulation of impurities with a calcium phosphate precipitate followed by the flotation of this coagulum to yield a clear liquor underflow. In recent years, a number of new materials[1-3,5] have been introduced in order to improve the efficiency of the process. An upgrading in the throughput of flotation clarifiers has been achieved by the application of high molecular weight anionic polyacrylamides as flotation aids.[5] Due to their long chain length and negative charge in solution, these polymers build up large flocs which can be floated at a much enhanced velocity. This ensures a high throughput of turbidity-free liquor.

One of the important developments has been the introduction of the Talofloc process[1-3] which aims at achieving a large measure of decolorization during defecation. A cationic surfactant (Talofloc) is added prior to the precipitation of phosphate and the final aerated suspension of calcium phosphate coagulum is flocculated by the addition of an anionic polyacrylamide (Taloflote). The advantage of obtaining considerable decolorization in the phosphatation process lies in the greater economy that can be achieved in the subsequent decolorizing stage.[2]

The purpose of this study was to investigate the degree of impurity removal that can be achieved with the aid of cationic flocculants. In addition, the effect on the process of a polyacrylamide flotation aid was investigated.

Experimental

The laboratory phosphatation unit and its mode of operation have been previously described, together with the experimental method for the determination of the filterability constant, K[4].

Cationic flocculants, used in this study, were Floccotan (a wattle extract in which the flavanoid components are polymerized by a Mannich reaction with formaldehyde and ethanolamine: molecular weight of about 50 000), Superfloc C573 and C577 (polyamine flocculants of molecular weight about 100 000), Nalcolyte 607, 8101, 4-WA-366 and 5-WP-549 (liquid polyamide flocculants of molecular weight about 100 000) and Superfloc C100 (a polyacrylamide of molecular weight greater than 1 million). Other flocculants employed were Superfloc N100 (a neutral polyacrylamide of molecular weight greater than 1 million) and Taloflote (an anionic polyacrylamide of molecular weight about 7 million[5]). The impurity removal achieved with these polymeric compounds was compared with that of the monomer Talofloc (diotadecyl dimethylammonium chloride). The above molecular weights are as published in the literature or were supplied by manufacturers' representatives. All flocculants were prepared in solution as recommended by their manufacturers.

Any chemical aid to the process can be applied either to the raw melt (prior to heating) or immediately before calcium phosphate precipitation or between the coagulum formation and flotation. The influence on impurity removal of the point of flocculant addition was investigated by the addition of Floccotan at each of the above positions. Similarly, a comparison was made of the efficiency of a flotation aid (Taloflote) applied both before and after the coagulation reaction.

A series of experiments was performed with varying amounts of cationic flocculants added immediately prior to the calcium phosphate precipitation in order to investigate their effect on impurity removal and on the filterability of clear liquor. The change in impurity levels with the use of Taloflote was studied by the addition of 5 ppm Taloflote after phosphatation of a melt which had previously been treated with either Floccotan, Superfloc C573 or Talofloc.

Since it has been shown[4] that starch has a deleterious effect on processing, this survey was concluded by observing the effect of a decolorizing flocculant (Superfloc C573) on a high starch melt. To achieve this high starch condition, an extra 100 ppm potato starch was added to the melt.

Results

The results of the experiments relating to the position of flocculant application indicated that the correct point for the addition of auxiliary defecants is dependent on the purpose for which they are required. Table 1 shows the results of adding 150 ppm Floccotan at three separate stages of the process.

TABLE 1
Comparison of different points for Floccotan addition

Point of Addition	Filt. Const. K	Clear Liquor Colour a * c at 420 nm	Clear Liquor P_2O_5 ppm
No Floccotan . . .	180	0,73	50
Floccotan in Pressure Vessel	250	0,68	45
Floccotan before precipitation . . .	140	0,68	30
Floccotan after precipitation . . .	180	0,73	50

Floccotan added immediately prior to phosphate precipitation produced the best overall result. Although addition before heating produced similar decolorization, it had the disadvantage of yielding a lower filterability and a greater phosphate content in the clear liquor as compared with flocculant addition immediately prior to phosphate precipitation. The application of Floccotan after precipitation did not have any effect on impurity removal and the results compared with the experiment in which no Floccotan was added. In none of these experiments did Floccotan act as a flotation aid.

If flocculant is to be employed as a flotation aid, the following results indicated that it must be added after the coagulation of calcium phosphate. For example, 5 ppm Taloflote added in this manner gave improved flocculation, large flocs and a rapid flotation, whereas if added prior to the phosphate precipitation, no improvement in flotation velocity was noticed. Irrespective of the point of addition, the scum obtained with the use of Taloflote tended to be thick and the foam head in the flotation vessel was greatly reduced (Table 2). Taloflote always gave the clear liquor a lower filterability although the addition of Taloflote subsequent to the coagulation reaction produced a lesser drop in filterability as well as a concomitant decrease in the phosphate remaining in clear liquor.

The enhanced removal of impurities obtained with the use of the flocculants listed in the Experimental Section was studied by adding various amounts (up to 600 ppm) to the

TABLE 2
Comparison of different points for Taloflote addition

Point of Addition	Filt. Const. K	Clear Liquor P_2O_5 ppm	Foam Head (Relative units)
No Taloflote	130	40	100
Taloflote before precipitation. . . .	370	45	81
Taloflote after precipitation	280	20	70

TABLE 3
Effect of 5 ppm Taloflote on impurities in clear liquor

	Colour a * c at 420 nm		Starch ppm		P_2O_5 ppm		Filt. Const. K		Foam Head	
Taloflote Dosage (ppm) . .	0	5	0	5	0	5	0	5	0	5
Decolorising Aid										
C573	0,78	0,78	60	55	35	30	200	340	100	78
Floccotan	0,80	0,82	65	55	25	25	190	290	100	73
Talofloc	0,55	0,57	45	45	40	20	130	280	100	70

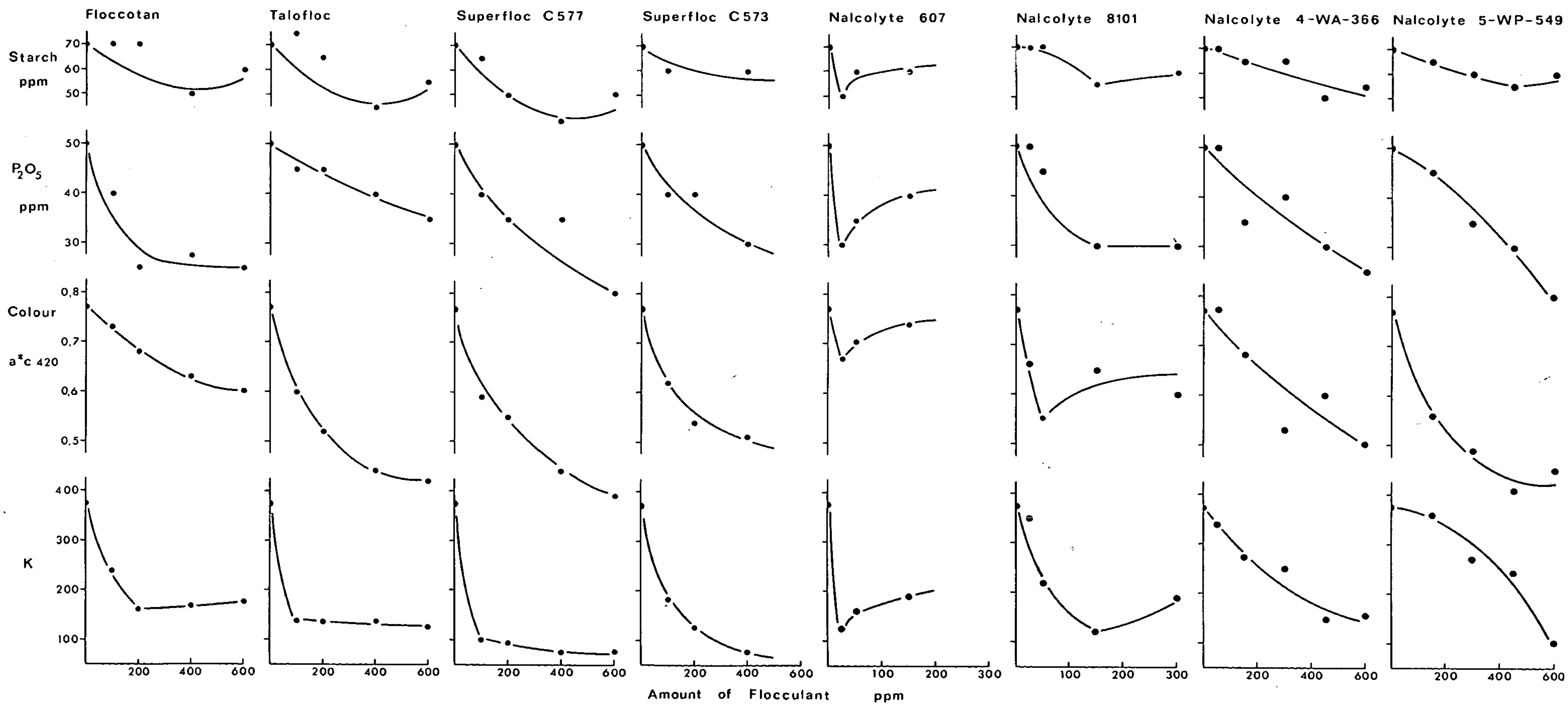

FIGURE 1 Impurity levels in clear liquor achieved with cationic flocculants added prior to phosphatation.

liquor immediately prior to the phosphatation reaction. The final clear liquor quality was assessed with respect to turbidity, conductivity ash, colour, phosphate content, starch content and filterability. The latter was expressed in terms of the filterability constant K, the meaning of which has previously been discussed.[4]

The conventional phosflotation process yielded liquor with low turbidity and it was found that the addition of flocculants had no effect on turbidity. However, it has been reported[5] that in situations in which there is an incomplete removal of turbidity, anionic polyacrylamide flocculants can improve clarity. In contrast to turbidity, the removal of ash in the conventional phosflotation process is negligible and, as expected, flocculants had no effect on this aspect.

The influence of flocculants on the other parameters of liquor quality depends on the nature of the flocculant and its applied dosage. The performance of different flocculants might also depend on the raw sugar quality and hence the following results may illustrate only general trends.

Figure 1 shows the performance of the cationic flocculants Superfloc C573, Superfloc C577, Floccotan, Nalcolyte 5-WP-549, 4-WA-366, 607 and 8101 compared with Talofloc. All these except Nalcolyte 607 and 8101 which were tested in the 0-300 ppm range, were studied with additions of up to 600 ppm on solids. The general trend of the results indicated an improvement in the efficiency of the phosphatation reaction as regards clear liquor quality. There was an enhanced removal of colour and starch as well as a reduction in the filterability constant and the P_2O_5 content of clear liquor. In all cases other than Nalcolyte 607 and 8101, the amount of colour removed tended to level off above 400 ppm dosage. Nalcolyte 607 gave slightly improved colour with a minimum at 25 ppm while Nalcolyte 8101 showed a corresponding minimum at 50 ppm. The reduction in P_2O_5 content of clear liquor tended to parallel the fall in the filterability constant with Nalcolyte 607 again showing a pronounced minimum at 25 ppm. All the flocculants and Talofloc showed some improved starch removal with a maximum effect generally around the 400 ppm level.

Although all these cationic flocculants produced beneficial results as regards clear liquor quality, none acted as a flotation aid by visibly enhancing floc formation and thus raising the flotation velocity. The foam heads produced in the flotation vessel were unaffected by flocculant usage.

Figure 2 shows the effect, on clear liquor quality, of applying the polyacrylamide flocculants Superfloc C100 (cationic), Superfloc N100 (neutral) and Taloflote (anionic) to the melt prior to the phosphate precipitation. These flocculants were studied in the 0-10 ppm range. Large doses ($\pm$ 50 ppm) had the effect of completely destroying the coagulation process so that no defecation or flotation occurred. Both the C100 and N100 flocculants produced simultaneous drastic increases in K and in the residual P_2O_5. There was a slight improvement in colour removal and no detectable increase in starch removal. An anionic flocculant such as Taloflote is not expected to act as a decolorising agent due to its negative charge in solution. The only effect that this flocculant had on liquor quality was a simultaneous increase in the filterability constant and the P_2O_5 content of clear liquor.

The group of anionic polyacrylamide flocculants (e.g. Taloflote) have found use as flotation aids.[5] In this regard, the above results showed that they must be added subsequent to the phosphate coagulation as otherwise they interfere with the defecation process and are unavailable thereafter for a secondary flocculation reaction. Table 3 indicates the changes in clear liquor quality resulting from the application of Taloflote to the phosphate coagulum formed in a melt which had

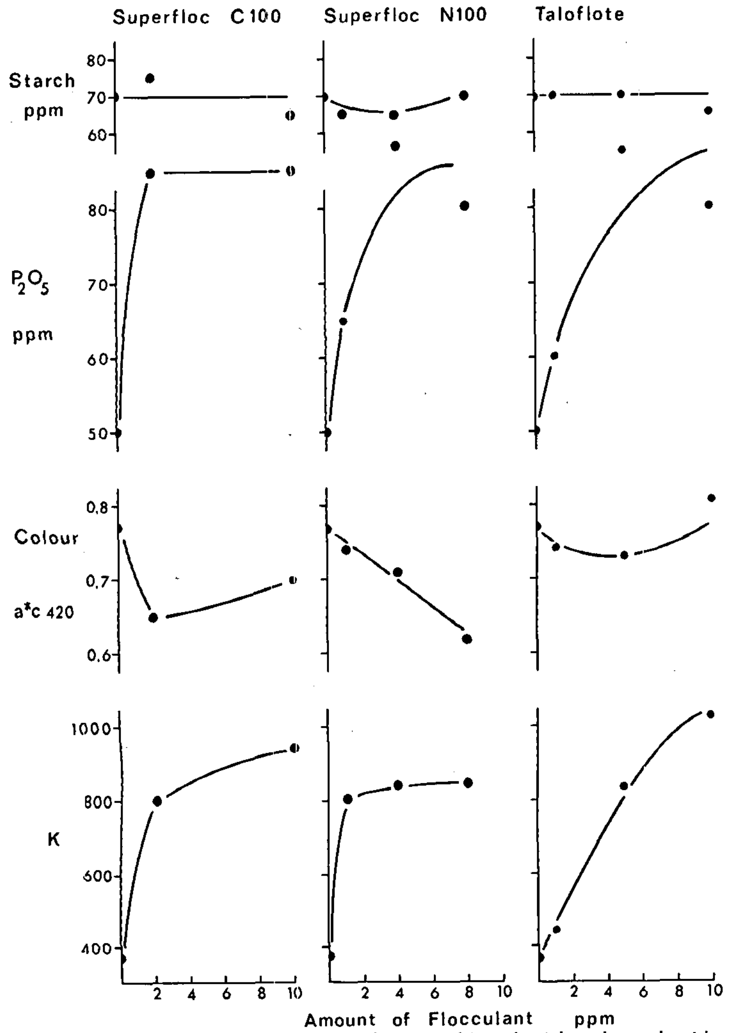

FIGURE 2 Impurity levels in clear liquor achieved with polyacrylamide flocculants added prior to phosphatation.

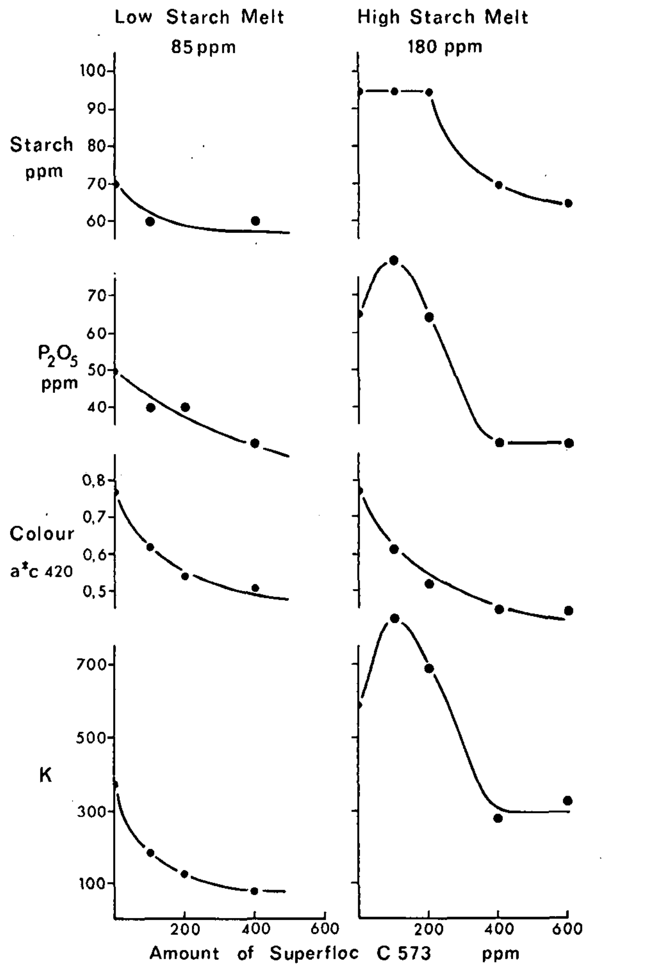

FIGURE 3 Comparison of a low and high starch melt with respect to impurity removal using Superfloc C573 as a chemical aid.

been treated with a cationic flocculant. The Taloflote acted efficiently as a flotation aid, decreased the foam head and, by its flocculating action, tended to lower the residual phosphate in clear liquor. It produced no improvement in liquor colour and had the disadvantage of decreasing the filterability.

The results of the experiments on a high in starch melt are contrasted in Figure 3 with those on the low starch melt. The most drastic change observed was in filterability and phosphate content of clear liquor. As previously shown[4] the presence of starch in the raw melt causes a retention of calcium phosphate particles in suspension. This results in a low filterability and considerable phosphate retention. The application of 100 ppm Superfloc C573 produced a deterioration in this aspect of the process with both filterability constant and P_2O_5 content showing sharp maxima. These two parameters then decreased in value to a constant level after the addition of 400 ppm of flocculant. At no stage, however, did the respective values approach those observed in the low starch experiments. The increased starch removal produced in the low starch case was suppressed until the P_2O_5 content of clear liquor began to show an overall decrease. The high starch melt produced a low foam head and a thick, viscous scum which returned to normal with the use of 400 ppm or more of flocculant. As regards colour, both low and high starch experiments produced similar results with the optimum dose at 400 ppm. Thus the removal of colour and phosphate and the increase in filterability were all achieved with an amount of 400 ppm flocculant which also removed the effect starch has on the scum and foam head.

Discussion

The effects produced by the flocculants studied can generally be understood in terms of the physico-chemical mechanism of the phosflotation process. This mechanism involves a coagulation of impurities (such as suspended matter and colour bodies) with the precipitated calcium phosphate. The liquor is then aerated and the coagulum floated to the surface in the form of a scum. Provided the process is run at a favourable air: solids ratio[4, 6], an almost complete removal of turbidity is achieved without the aid of flocculants.

The removal of colour can be improved by use of the Talofloc process[1-3] in which the anionic colour bodies are rendered insoluble by the addition of a cationic surfactant. This is an ionic interaction and the above results indicate that certain cationic flocculants can act in an analogous manner. Due to its electrostatic nature, colour destabilization is a rapid process and, provided efficient mixing is achieved, the flocculant can be added immediately prior to the other defecants. The produced colour body — surfactant complex is readily incorporated into the calcium phosphate coagulum. The addition of cationic flocculant after the precipitation of calcium phosphate appeared to have no decolorizing action since the impurity scavenging effect of the precipitation reaction was then lost.

It has been shown[4] that certain impurities (e.g. starch) can act as a protective colloid and stabilize colloidal phosphate particles in suspension. Although these particles do not contribute measurably to the turbidity, their presence causes a rise both in the filterability constant, K, and in the residual phosphate in clear liquor. The results relating to the application of polyacrylamide flocculants both before and after phosphate precipitation (Table 2 and Figure 2) can be interpreted by postulating that the polyacrylamide molecule (molecular weight greater than one million[5]) is of sufficient size to behave in a manner analogous to starch. Thus the presence of polyacrylamides prior to phosphatation results in the stabilization in suspension of calcium phosphate crystallites on whose surface the long chain flocculant molecule has collapsed. This would account for the high phosphate levels and K values observed in Figure 2. Once the polyacrylamide molecule has collpased onto the particle surface, it will be unavailable for any subsequent flocculation reaction and thus does not act as a flotation aid. It is therefore imperative to add a flotation aid such as Taloflote after the primary phosphate coagulation. In this situation, the polyacrylamide can bind together the primary coagulated particles to form a stable floc which can be floated at a much enhanced velocity. Thus the flocculant acts as a scavenging agent for the precipitate and produces a significant reduction in the P_2O_5 content of clear liquor (Table 3).

The decrease in filterability even when Taloflote is added in the correct manner appears anomalous. That this is not due to residual flocculant was shown by conducting a number of filtration runs in which varying amounts of Taloflote, added to a refined sugar melt, produced no effect on K. Possibly the decrease in filterability is due to some colloidal material other than starch which is not incorporated into the coagulum because of the rapid flocculation induced by Taloflote.

The action of cationic flocculants and Talofloc (Figure 1) in decreasing starch and the filterability constant of clear liquor would appear to be linked with the decrease in P_2O_5 concentration. Micro-electrophoretic measurements indicated that the calcium phosphate particles precipitated in the liquor are anionic and it may thus be surmised that cationic additives produce a charge destabilization so that the suspended particles are more readily incorporated into the coagulum. In addition, any starch associated with such a destabilized particle will also be removed. Thus the reduction in starch and P_2O_5 content of clear liquor may both be ascribed to an improved coagulation and flotation effected by cationic surface active agents which aid in the destabilization of suspended particles.

Further evidence for the assumption that enhanced starch removal is associated with the decrease in residual phosphate can be seen in Figure 2 which shows that the polyacrylamide flocculants which produce no phosphate reduction also yield a constant starch concentration in clear liquor. Similarly enhanced starch removal is only observed in the high starch experiment shown in Figure 3 when there is an overall reduction in P_2O_5 concentration.

Conclusion

This study has amplified the advantages that can be obtained by the use of auxiliary defecants in the phosflotation process. The results can be briefly summarized as follows:

(1) In order to achieve improvements it is essential to add the appropriate auxiliary defecant at the correct stage of the process.
(2) Certain cationic chemical aids produce considerable decolorization. These chemical agents also improve phosphate and starch removal and raise the filterability of clear liquor.
(3) The application of flocculants (in the correct amounts) can remove to some extent the disadvantageous effects of starch.

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