

CATIONIC POLYMERS HELP SEPARATE PRELIMER SUSPENDED SOLIDS

Lloyd W. Norman, Edward W. Gannon, John B. Pratt and John W. Davey
PPG/Mazer Chemicals, 3938 Porett Dr., Gurnee, IL 60031

INTRODUCTION: For many years it has been well known that actual physical removal of the suspended solids generated in the prelimer results in a final purified juice of superior quality. Unfortunately, the solids generated have proven difficult to remove. To our knowledge no commercially successful technique is presently in use within the United States, although at one point in time the Amalgamated Sugar Company successfully employed their "TASCO" system commercially.

We believe that the use of certain cationic polymers may soon make the separation both achievable and economically attractive in modern beet sugar plants. Experimental testing on both the laboratory and plant scale has demonstrated improved settling and improved filtering properties of prelimer suspended solids at various levels of polymer addition.

Anionic polymers have been used at least since the 1950's to enhance the settling and filtration of the suspended solids encountered in First Carbonation. It is doubtful that any modern beet sugar plant would consider operating without these polymers. The Enviro-Clear clarifier was specifically designed to use anionic polymer flocculants. However, the anionic's have not proven to be useful in settling and filtering the prelimer solids.

Our initial use of cationic polymers came about because of a recent campaign in which large tonnages of beets in several areas were damaged by early freezing. As a result, the beets deteriorated excessively in storage, and processing problems, such as poor 2nd Carb filtration, were encountered. When the addition of cationic polymer demonstrated improved 2nd Carb filtration in the Lab, successful plant utilization followed.

Because the filtration difficulties at 2nd Carb were attributed by some to the presence of excessive colloidal material, and because stabilization of colloids occurs in the prelimer, it follows that cationic polymers might be used to assist in the removal of prelimer suspended solids. Hence the work being reported here.

The cationic polymers used in this work were dimethylamine-epichlorohydrin copolymers approved by the FDA at the maximum level of 150 parts per million based upon the weight of sugar in the treated process stream. Since the polymers are marketed as 50 percent solutions in water, the clearance level becomes 300 ppm. They are available in different molecular weights. Our work indicated that the medium to high molecular weight products performed best. We chose the medium molecular weight for the majority

of our work because the viscosity of the solution allowed direct metering of the "neat" 50% solution. Additionally, laboratory tests indicated equal or better performance than the higher molecular weight (dilution required for metered pumping). Since polymer effectiveness is highly dependant upon the particular juice substrate, selection of the best performer must be made for each individual plant.

Calculation of the maximum amount of 50% polymer solution that can be added gives about 30 ppm for a juice stream of about 10% sugar concentration. On a volume/volume basis this means addition of 30 gallons of the polymer solution for every million gallons of treated juice. It should be pointed out that these values are approximations only and that the allowable addition should be verified for the each and every individual application.

METHODS: Without the cooperation and assistance of people at the beet plants where our work was done, this report would not be possible. All work was on site at operating plants. Please note the acknowledgment appended hereto.

Before work was attempted on the actual plant juice streams laboratory tests were made to establish some of the operating parameters for plant tests. Grab samples of preliner overflow were treated with cationic polymers at various levels of addition. Because it is virtually impossible to duplicate the plant filtration conditions on a laboratory scale, a simple gravity filtration procedure was devised. The results are not intended to claim reproducibility by plant filters. They are, however, comparative one with another and are believed to be directly related to plant filter operation.

The procedure used was as follows: To each of a series of 1000 ml beakers 500 ml of the slurry to be tested was introduced (usually either preliner overflow, concentrated preliner sludge or 1st Carb sludge). All samples were "grab" samples. The desired levels of polymer treatment were added, with an untreated blank included in most runs. After treatment the samples were stirred for a minimum of 3 minutes (found to be the minimum for reasonable consistency of results); then, 150 ml of well mixed sample was withdrawn and filtered by gravity through Whatman No. 2 filter paper. The filtrate was collected in a 100 ml graduated cylinder. Filtrate accumulation was timed starting the instant the juice touched the filter paper. Stop watch readings were recorded at 5 ml, 10 ml, 15 ml and 20 ml, etc. The effect of polymer addition was judged by comparison of graphical plots of filtration rate vs filtrate volume for the various tests, as will be shown and discussed later.

In some of the later series of tests an Eimco filter leaf was used in filtration testing. The leaf has 0.1 square foot of filter surface. Industrial filter cloth circles of any desired type can be used in the apparatus. In our procedure the leaf was hooked up to a vacuum

supply maintained at 13 inches of mercury to approximate rotary vacuum filter conditions. The leaf was submerged for one minute in a stirred sample of the slurry being tested. After one minute the leaf was lifted from the slurry and the filtrate was allowed to drain into the vacuum receptacle for measurement of the filtrate quantity. Just as with the gravity tests, comparative filtration rates were used to judge the test results. With the known filter area rates per square foot could also be calculated and compared.

Settling tests were conducted by placing 500 ml. of well mixed slurry in a 500 ml. graduated cylinder and noting the time required for the interface between the settling sludge mass and the clear supernatant to pass the volume marks on the graduated cylinder (450, 400, 350, etc.). Graphing of the time of settling versus the interface level generates curves that can be used in judging the effectiveness of treatment.

RESULTS AND DISCUSSION: The first order of business was to determine the effectiveness of the cationic polymer, so that levels of desired usage could be determined. In Figure 1 the filtration rate of preliner overflow is shown for treatment levels of 0 to 30 ppm. As might be expected, the rate increases with each incremental increase in polymer treatment. Note that the incremental increase from 20 to 30 ppm gave better improvement than that from 10 to 20. This was not expected, although the same anomaly was encountered at more than one of the sugar plants. See Figure 1.

It was shown that even higher addition rates gave still higher rates of filtration. In Figure 2 polymer additions of 0, 30, and 100 ppm are shown. The added amount over 30 ppm does further increase the rates obtained, but at incrementally less effectiveness, as might be expected. Rates as high as 300 ppm continued to improve filtration; however, unless greater clearance from the FDA were to be obtained, anything over about 30 ppm is academic. See Figure 2.

Because anionic polymers are already in use at sugar plants, comparison of anionic versus cationic for preliner treatment was desirable. The normal addition level of anionic polymer to 1st Carb juice is usually in the 3 to 5 ppm area. We chose to compare 5 ppm of a widely used anionic with 30 ppm cationic and with a couple of mixtures of the two. The results shown in Figure 3 clearly demonstrate that the anionic used was not effective. It is further shown that mixing a fraction of anionic with the cationic depresses the activity of the cationic. While not shown in the figure, it was found that the addition of the anionic alone depressed the filterability of untreated preliner slurry. See Figure 3.

If the suspended solids are to be successfully separated from preliner effluent, it stands to reason that conventional settling and filtration might be the initial technology of choice. However, other removal techniques such as liquid cyclones, belt filters, or the new automated plate and frame filters should be considered. Lab tests of the effect of cationic

polymers on settling rates were run, as reported in Figure 4. Some improvement is demonstrated. It should be noted that anionic polymers do a better job of settling enhancement than the cationic, either alone or in combination with the cationic. During one plant test anionic polymer at the rate of 1/2 ppm was added to the plant stream. Unfortunately no comparative data could be obtained. Efforts are in order to optimize the best properties of each while minimizing adverse contribution. We intend further work. See Figure 4.

Ideally in any experimental investigation all of the variables other than those being evaluated should be held constant. In the laboratory this situation can be approached. However, in plant tests the natural variability of the raw material, along with problems in both process control and equipment performance, combine to cause variability in conditions and erratic experimental results. Replication of tests and extended testing periods help, but do not eliminate the difficulties. In our work the usual flow variations, equipment troubles, etc., were encountered, as will be discussed later.

From laboratory work it was established that the best results were obtained with maximum allowable use of the cationic polymer, so in nearly all instances a rate of 30 ppm was employed in plant trials. The point of addition can be critical in polymer applications. In the earliest tests we tried polymer addition to cells 1 and 3 of the 6 cell preliher, also to the effluent overflow from cell 6. Our conclusion was that cell 1 was the best choice. In later work addition to raw juice after the heater was also tried. Relatively little difference could be detected in the results. From one perspective addition should be as early in the process as possible to give plenty of time for reaction and for polymer to be present at each of the progressive iso-electric points for individual colloids. Another perspective would suggest having fresh polymer present to stabilize particles of suspended solids at the point of settling and filtration. We believe that this issue is not settled and more work should be done to find the optimum injection point. It could well be that the best results may be obtained by splitting the polymer addition, perhaps to the extent that some goes into each of the preliher cells.

In one plant test data was obtained over a ten day period with 30 ppm of cationic polymer being added during five of those days. No treatment was used the day before and the four days after the trial period. At this plant separation of a concentrated preliher effluent is practiced. During the test the preliher concentrate was sent directly to 1st Carb, bypassing the main limer. Filtration measurements using the Eimco vacuum filter leaf were made at least once each shift (twice when possible) on both the preliher sludge (concentrated effluent) and 1st Carb sludge (clarifier underflow). In Table 1 below the results of these tests are given. The first column gives comparison of the overall average of treated versus untreated rates of filtration. All rates are in gallons per minute per square foot of filter surface. The next two columns give the same comparison showing

the range of the average data for the days with the highest and lowest rates for each category.

Table 1
Filtration Rates as GPM per square foot
for Prelimer and 1st Carb Sludge

Sample Filtered	Overall Average Rate	Rate Range Day Average	
		High	Low
Prelimer--Untreated	0.16	0.23	0.13
" " 30 ppm	0.23	0.26	0.19
1st Carb--Untreated	0.38	0.42	0.27
" " 30 ppm	0.44	0.47	0.39

It is interesting to note that in the overall average column addition of 30 ppm of cationic polymer gave a 44% increase in the filtration rate for the Prelimer sludge. The increase for 1st Carb sludge was 16%. This indicates a good carryover of the effect of polymer to the 1st Carb station. Comparison of high days with low days demonstrates the wide fluctuation in results encountered in plant testing. But, it is also interesting to note that the range of data was lessened by polymer treatment in the case of both Prelimer and 1st Carb sludge. Also the variability is less with 1st Carb than with Prelimer results. Overall, the ratio of 1st Carb rate to Prelimer rate was reduced from nearly 2.5 : 1 to less than 2.0 : 1 by the polymer treatment.

Settling rates for Prelimer effluent and 1st Carb slurry resulting from this test are depicted in Figures 5 and 6. A look at these graphs will show that polymer treatment was not beneficial on the average, although the difference for 1st Carb was not great. This was unexpected, since laboratory tests tended to show some benefit from treatment. See Figures 5 & 6.

The undesirable inconsistency of plant data is demonstrated again. The variability between the best and worst days average is rather wide and would be even wider if individual best and worst determinations had been used. As with the filtration rate comparisons, the treatment reduced the range of variability. This was particularly true for the settling rates for 1st Carb (see Figure 6) where a fairly tight range was found.

To demonstrate the effect upon plant filtration, if any, of mixing concentrated Prelimer sludge with normal 1st Carb sludge one of the rotary vacuum filters was isolated and fed with the mixture. The percentage of Prelimer sludge was gradually increased until the volume ratio was 30% Prelimer and 70% 1st Carb. The filter was run for several hours with this mixture and for a short time with mixtures approaching 100% Prelimer sludge.

No problems were encountered with filtration rate, cake discharge or sugar loss, although the time of operation was necessarily brief.

A few laboratory determinations indicated that the level of suspended solids was about 6 to 7 times greater in the 1st Carb sludge than the Prelimer sludge. This is in the neighborhood of the ratio of CaO added to each. The ratio of juice nonsugars should be much higher in the Prelimer suspended solids, should contain a higher organic component, and logically be fluffier and lighter. Hence, the difficulty always encountered in attempting to filter this slurry.

In 1st Carb sludge the extra CaO gives added weight and bulk to the suspended solids allowing economical settling and filtration. When transferred to the prelimer, this weight and bulk should provide similar filtration. This can be done only by "closing the loop". To do this:

1. The Prelimer suspended solids must be physically removed.

2. The 1st Carb clarifier underflow must be recycled to the prelimer.

This will send relatively clean juice to 1st Carb and the recycled solids will be nearly all freshly precipitated, highly surface active calcium carbonate. Both the added bulk of the CaO and the additional surface activity should be helpful in settling and filtering operations. Hopefully, closed loop operation can soon be attempted and proven. Until then this new technology will not have been satisfactorily demonstrated, nor will all of the anticipated advantages have been realized.

Our work has shown only promise of success. Much more work is needed. In all of the plant trials conducted there has been the feeling that qualitatively the juice is improved by the addition of 30 ppm of cationic polymer. Plant personnel have said that the color and clarity are better. However, our hopes for numerical proof have not materialized, whether because of process variability or failure of the basic premise. The hints are there, but not the proof--yet.

In this work the stakes are extraordinarily high. Few would disagree, it is believed, that successful Prelimer solids removal will yield better quality juice and the economic advantages associated therewith. To get the job done the loop must be closed. In Figure 7 a schematic flow diagram is given. See figure 7.

Three major deviations from conventional operation are indicated:

1. A clarifier and filters are provided after the Prelimer (the filters can be those now used for 1st Carb filtration.

2. Recycle lines to return all 1st Carb clarifier underflow to the Prelimer system.

3. Addition of cationic polymer.

The major capital cost would be the new clarifier and to be on the safe side perhaps an extra filter would be desirable. Recycle lines for 1st Carb sludge would not be a major cost. The plant could be started up in the conventional manner. Then one filter could be isolated for Prelimer sludge and a small amount of this material could be sent to that filter with a corresponding amount of recycle returned from 1st Carb. Incremental increases of Prelimer sludge to filtration and 1st Carb sludge return could be made. As each incremental amount became routine, another could be instituted and the loop could be gradually closed.

The major capital cost would be the added clarifier. Perhaps additional filter capacity would be needed. Changes in piping would not be major cost. When one calculates potential economic gain, the capital costs seem low relative to the potential returns. We hope this work can proceed to the eventual acceptance of the concept and to the financial advantage of everyone in the beet sugar industry.

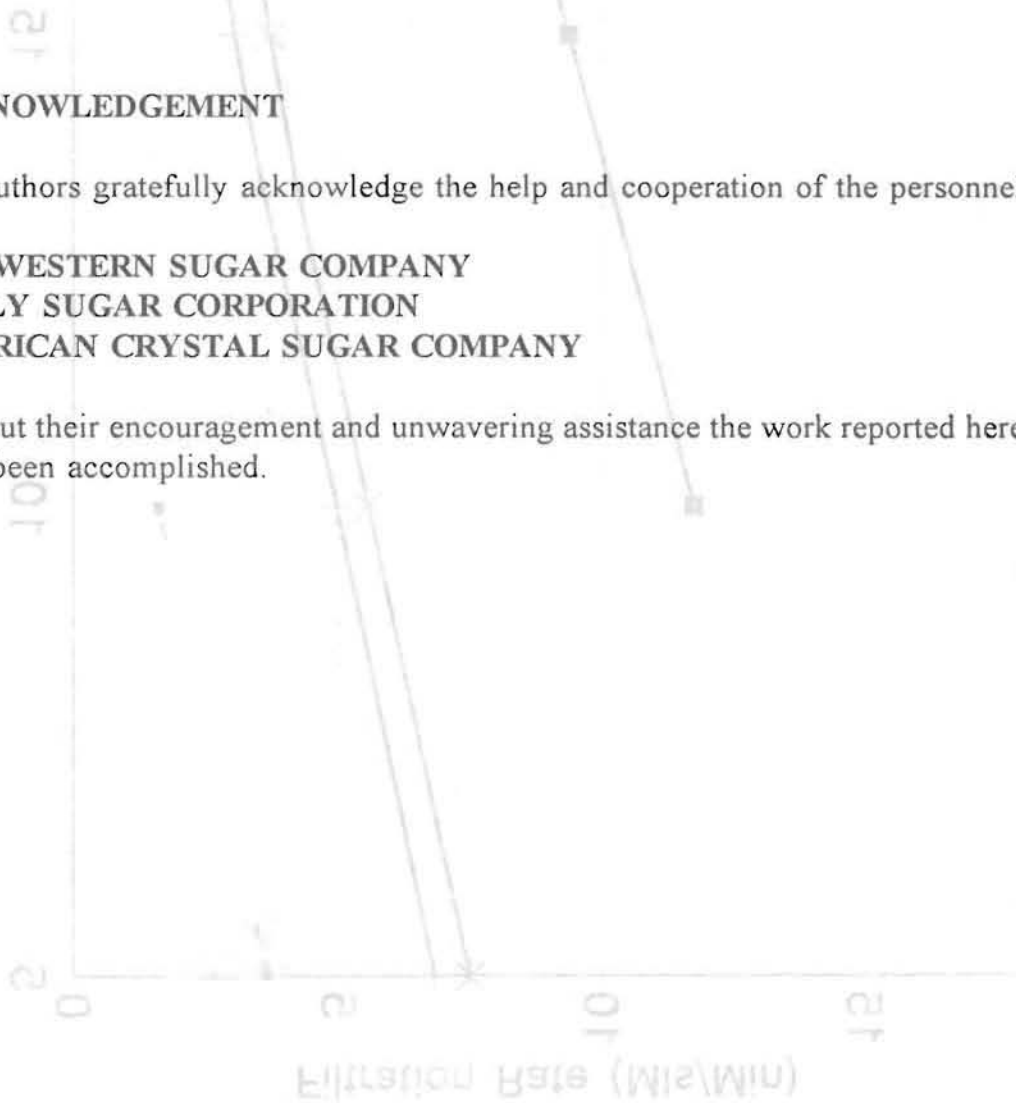
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Figure 1



The Effect Cationic Polymer on 1st Filter Rates

The Effect Cationic Polymer on Lab Filter Rates

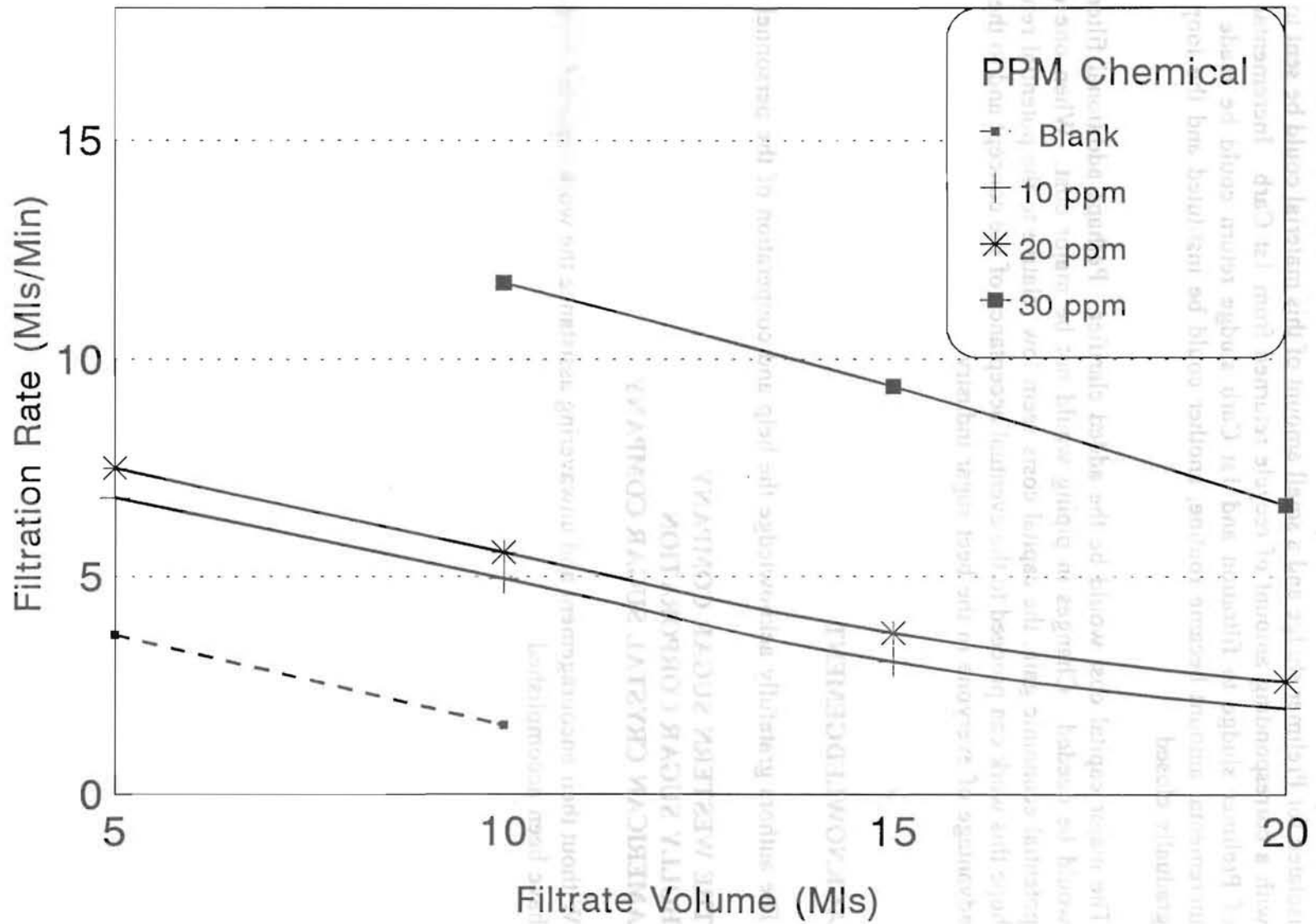
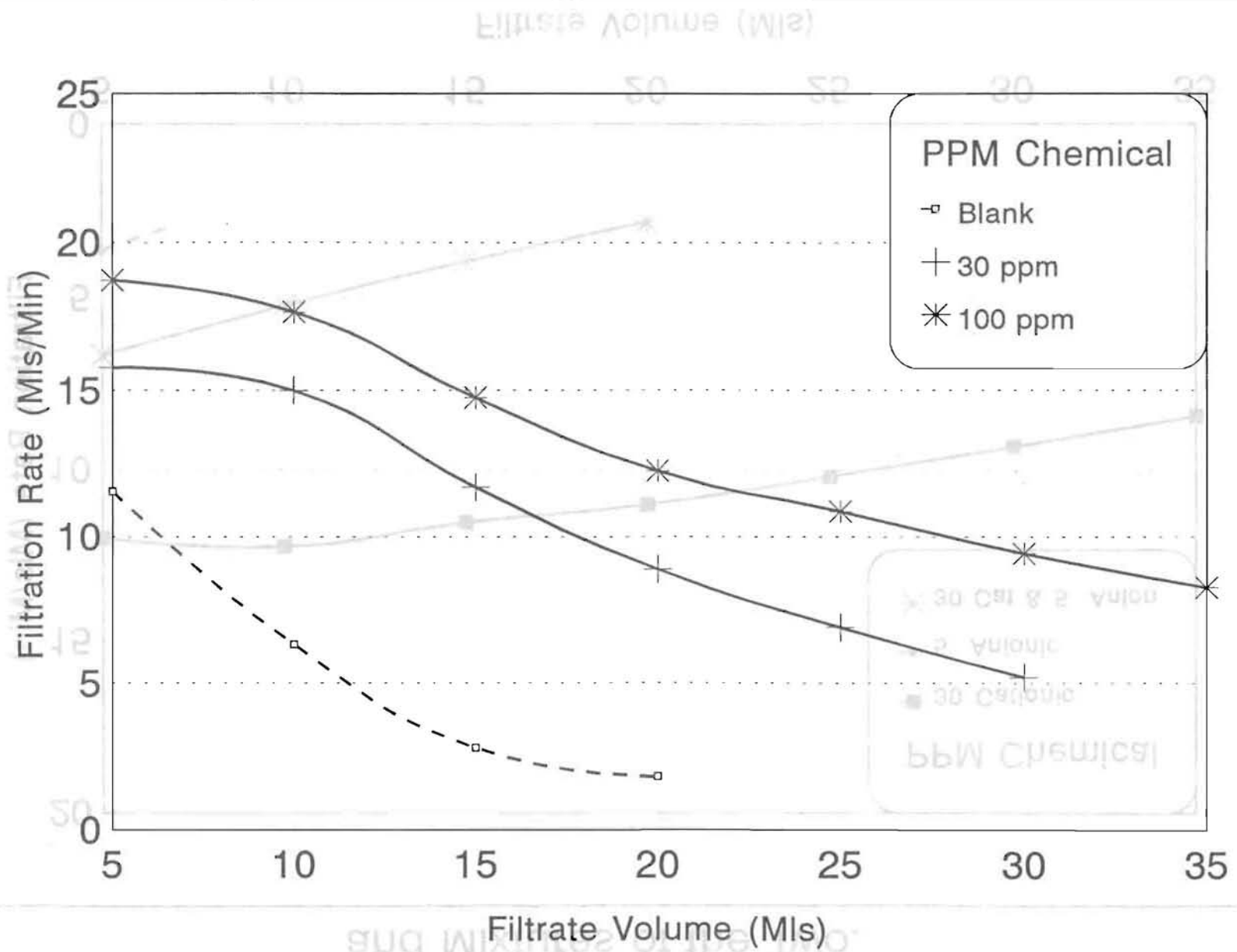


Figure 1

The Effect of Large Dosages of Cationic Polymer



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Figure 2 Comparison of Cationic with Anionic Polymer

Comparison of Cationic with Anionic Polymer and Mixtures of the Two.

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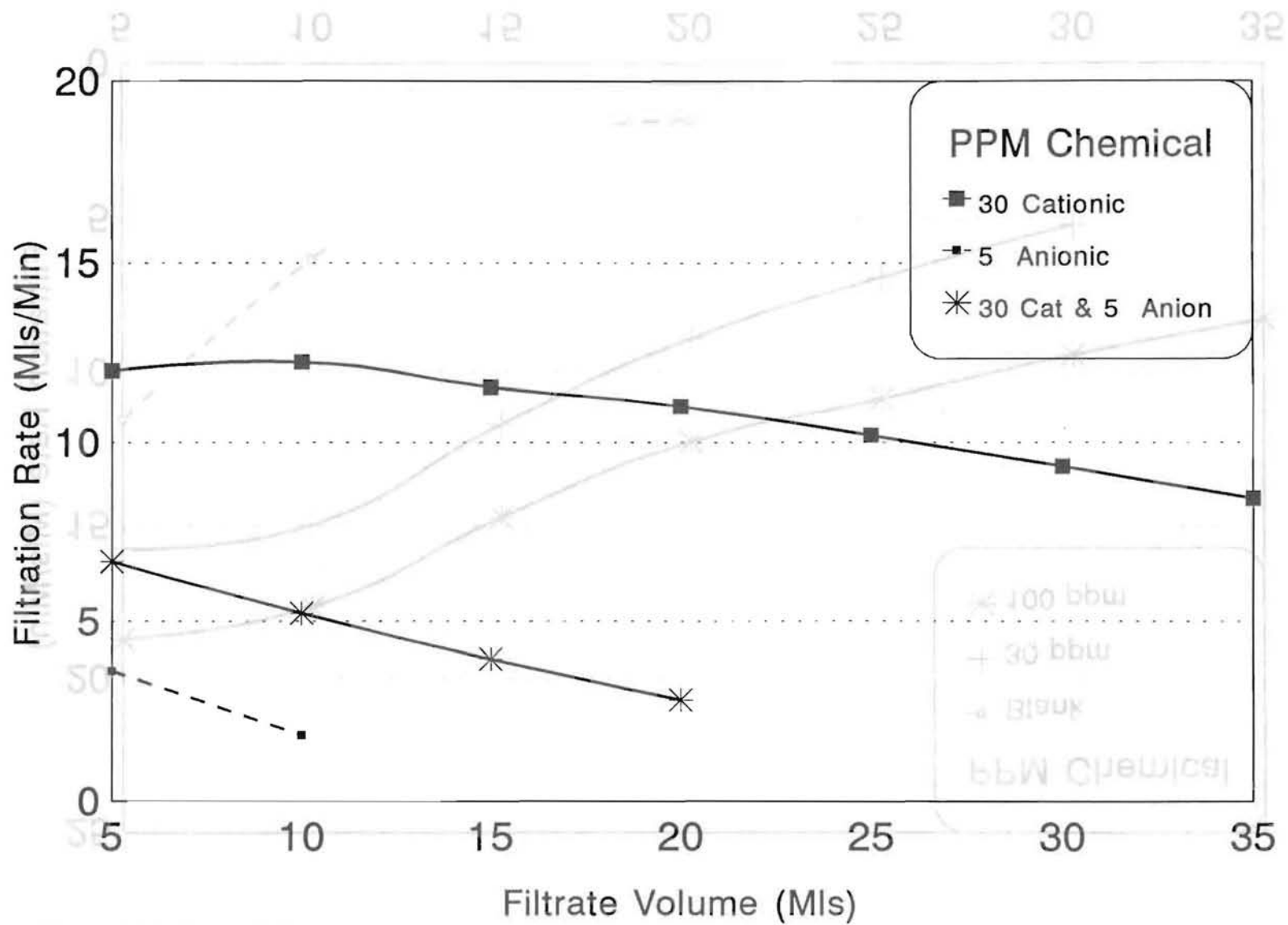
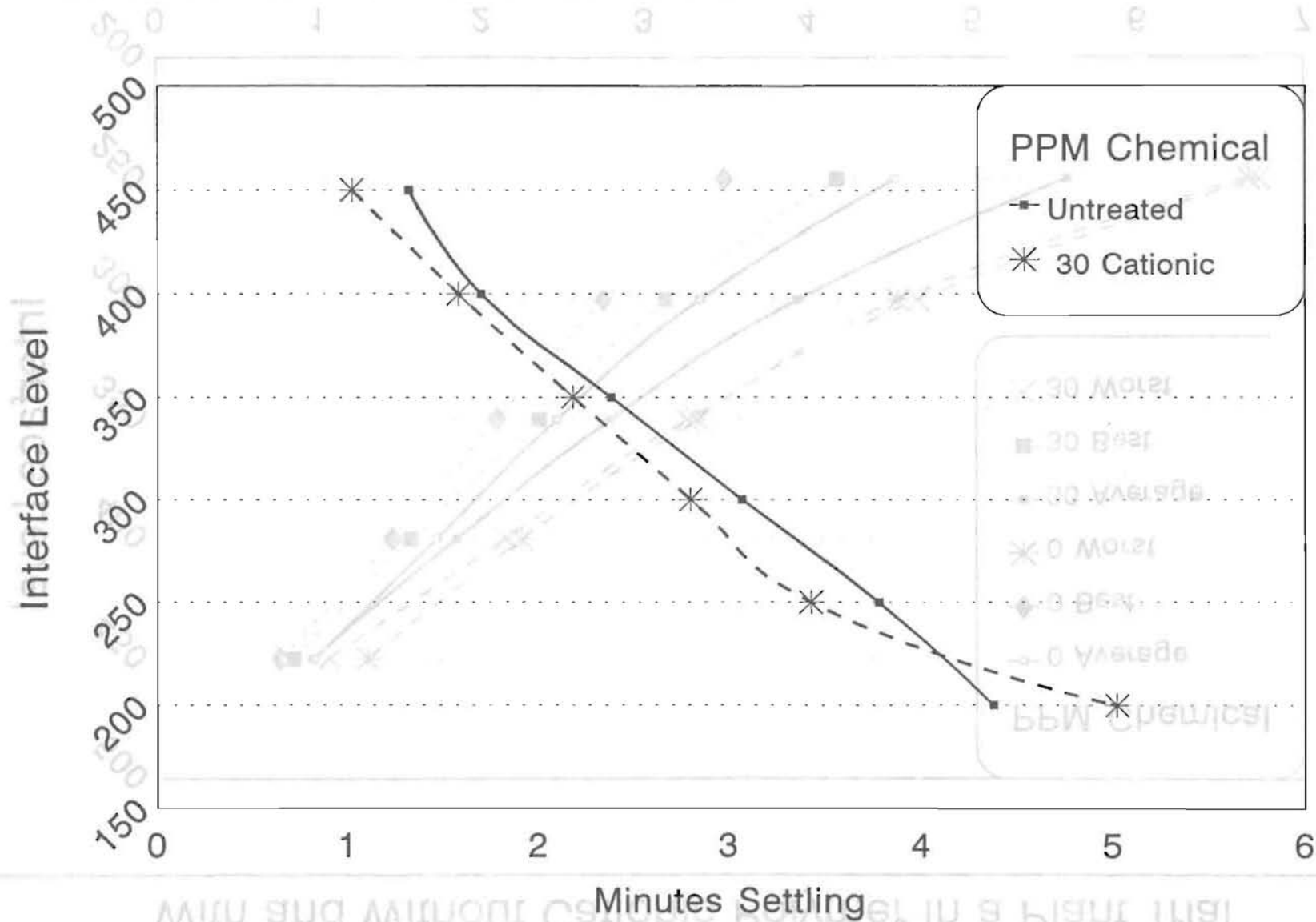


Figure 3

Effect of Cationic Polymer on the Settling Rate of Prelimer Suspended Solids

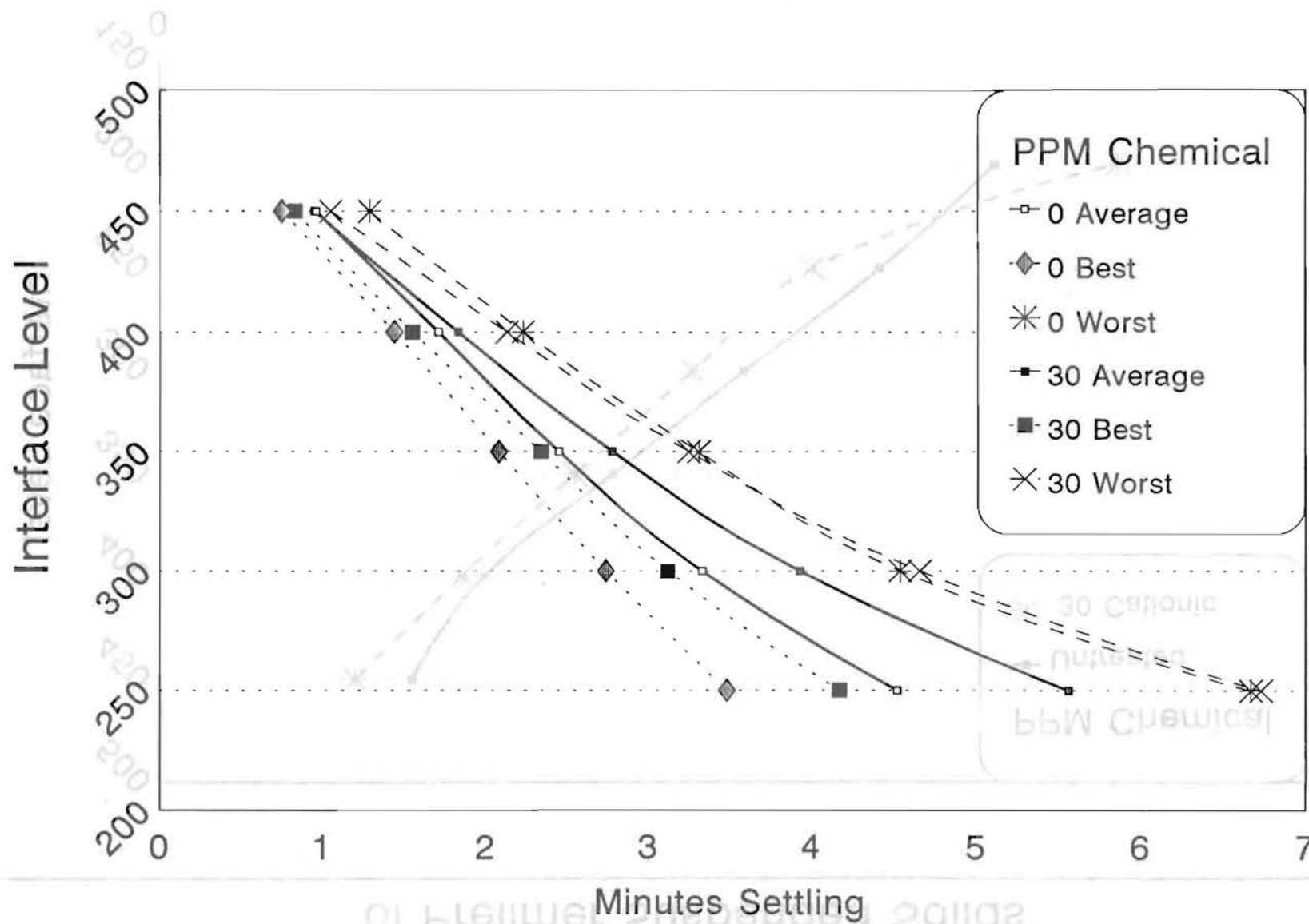


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Comparison of Settling of Prelimer Suspended Solids With and Without Cationic Polymer in a Plant Trial
Figure 4

Comparison of Settling of Prelimer Suspended Solids With and Without Cationic Polymer in a Plant Trial

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Effect of Cationic Polymer on the Settling Rate of Prelimer Suspended Solids
Figure 5

First Carb Settling Cationic Comparison in a Plant Trial

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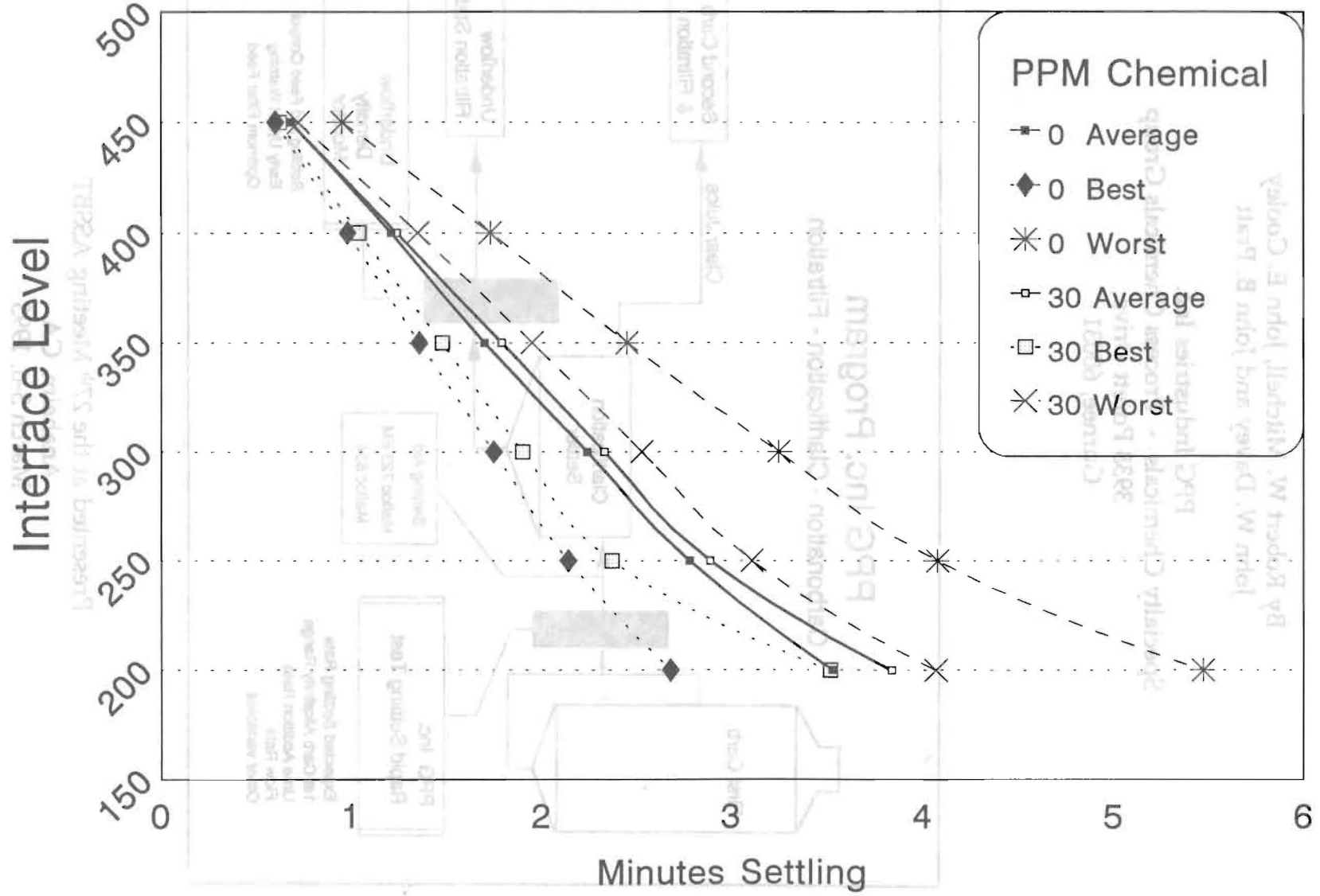


Figure 6