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September 1, 1989

Ms. W. F. Perrin, Technical Information Officer
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J. A. Duschinski
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I. DETAILS OF REQUEST FOR RELEASE

WSRC-RP-89-792, "PREDICTING THE PERFORMANCE OF CERAMIC FILTERS BY THE USE OF SILT DENSITY INDEX," By S. M. Cain.

A paper proposed for presentation at the South Carolina Junior Academy of Sciences and the CSRA Science Fair in April 1990.

Technical questions pertaining to the contents of this document should be addressed to the author(s) or

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II. DOE-SR ACTION

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___ Approved upon completion of changes marked on document.

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Date 9-15-89

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WSRC-RP-89-792

PREDICTING THE PERFORMANCE OF CERAMIC FILTERS BY THE USE OF SILT DENSITY INDEX (U)

by

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A paper proposed for presentation at the
South Carolina Junior Academy of Sciences
and the CSRA Science Fair
April 1990

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Advisory Scientist

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PREDICTING THE PERFORMANCE OF CERAMIC FILTERS BY THE USE OF SILT DENSITY INDEX (U)

by

Steven M. Cain

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ABSTRACT

A series of experiments was conducted to study the rate of filter fouling of several simulants. These solutions contained hydroxides of iron, aluminum, silica, and zinc, using a range of concentration of each under various conditions. Total Solids (TS), Total Suspended Solids (TSS), Total Dissolved Solids (TDS), Silt Density Index (SDI) , and conductivity were measured in the studies. The purpose of these experiments was to find out whether or not SDI could be used to accurately predict the performance of ceramic filters. The preliminary results show that SDI alone cannot be used, but can be used in conjunction with the total filtration time. The procedure is limited and can only be used for preliminary screening.

INTRODUCTION

Silt Density Index (SDI) has long been used to predict performance of ultrafilters (UF) and reverse osmosis (RO) membranes. The SDI measures a "rate" of fouling of a dead-ended filter. This has been seen to be indicative of membrane performance[1]. This has become increasingly important with the development of new RO and UF membranes, specifically the thin film composite (TFC) membranes. Pretreatment chemistry is often determined based upon a SDI or related experiment. Normally, the source of water for a given UF/RO system is rather constant.

Microfilters constructed of alpha-alumina, or ceramic, have recently received widespread attention. These filters are composed of layers of alumina in various pore sizes. The finest layer achievable utilizing only alpha alumina is 0.2 microns nominal pore size. This finest layer is formed into a thin membrane only 15 microns thick. Use of microfilters (MF) constructed of alumina has been limited to gas separations, wine filtration, and fruit juice filtration[2]. The treatment of aqueous streams in water treatment plants is a new application for ceramic filters. Advantages of ceramic filters include their long lifetime, chemical resistivity, and thermal stability. Product water quality is expected to be quite high due to the tight pore size distribution of the filters[3]. Any variability in water treatment plant influent could require different pretreatment chemistry. It would be advantageous to be able to identify water characteristics so that adequate pretreatment can be done and filter fouling can thus be avoided. This communication deals with the identification of various simulant solution parameters and compares them to actual filter performance curves.

The simulants used contain iron, silicon, aluminum, and zinc. The iron, aluminum, and zinc are added as the nitrate salts to nitric acid solutions. The silicon is added as sodium metasilicate to the nitric acid solutions. As these solutions are pH adjusted to near pH = 7.5, the metal hydroxides are expected to form and polymerize, and the silicon is expected-to coprecipitate as colloidal silica.

EXPERIMENTAL

The samples were used not only in SDI studies, but also in Total Solids(TS), Total Suspended Solids(TSS), and Total Dissolved Solids(TDS) experiments. The following is a description of each test:

SDI (Refer to Schematic 1): Insert the feed and bypass tubes into the sample container and insert the filtrate tube into the sample container. Insert a 0.45 micron Millipore filter into the in-line filter holder. Start the pump and adjust the pressure to 30 psi. Remove the filtrate line from the feed tank and measure the time required to filter 500 mL of solution. This number is the Initial Time (Ti). Continue to filter for 15 minutes. After this 15 minutes measure the time required to filter 500 mL of solution. This number is the Final Time (Tf). The Total Time = Ti + Tf. To calculate the SDI, use the following equation:

$$\mathrm{SDI} = \frac{(1\text{-}Ti/Tf) \times 100}{15 \text{ min}}$$

The range of values on the 15 minute SDI extends from 0 (low fouling potential) to 6.6 (severe fouling potential).

TS: Weigh a clean 250 mL beaker. Pipet 100 mL of solution into the beaker and evaporate to dryness. After evaporation, weigh the beaker and the residue.

TSS: Install a preweighed 0.45 micron Millipore filter in the filtering flask and carefully pipet 100 mL of solution through the filter. Desiccate and weigh the filter and the residue.

TDS: Add the filtrate from the TSS to a preweighed 250 mL beaker. Evaporate and weigh the beaker and the residue.

The area process water (fpw) was prefiltered to 5.0 micron (samples 8, 9, 8911,8914, 8917, 8918). D-tank water is a simulant that was prepared from $Fe(NO_3)_3$ and other salt solutions. All solutions and reagents were prepared from deionized water unless otherwise noted. Reagent grade $Fe(NO_3)_3 \cdot 9H_2O$, $Na_2SiO_3 \cdot 9H_2O$ (metasilicate), $Al(NO_3)_3 \cdot H_2O$, $Zn(NO_3)_2 \cdot 6H_2O$, HNO_3, NaOH, and Na_2CO_3 were used in all experiments. The solutions were prepared by dissolving the reagents in 0.1 M HNO_3. All solutions other than sample 8 and the D-tank solutions were titrated to pH = 7.5 ± .5 with either NaOH or Na_2CO_3.

Sample 1.	A solution (7.0 L) of 0.440 g of $Fe(NO_3)_3 \cdot 9H_2O$ was titrated with NaOH solution.
Sample 2.	A solution (7.0 L) of 0.237 g of $Fe(NO_3)_3 \cdot 9H_2O$ was titrated with NaOH solution.
Sample 3.	A solution (7.0 L) of 0.109 g of $Fe(NO_3)_3 \cdot 9H_2O$ was titrated with NaOH solution.
Sample 4.	A solution (7.0 L) of 0.3606 g of $Na_2SiO_3 \cdot 9H_20$ and 0.2907g of $Fe(NO_3)_3 \cdot 9H_2O$ was titrated with NaOH solution.

Sample 5.	A solution (7.0 L) of 0.3588 g of $Na_2SiO_3 \cdot 9H_2O$, 0.2892 g of $Fe(NO_3)_3.9H_2O$, and 0.4899 g of $Al(NO_3)_3 \cdot H_2O$ was titrated with NaOH solution.
Sample 6.	A solution (7.0 L) of 0.3579 g of $Na_2SiO_3 \cdot 9H_2O$, 0.2848 g of $Fe(NO_3)_3 \cdot 9H_2O$, 0.4863 g of $Al(NO_3)_3 \cdot H_2O$, and 0.0784 g of $Zn(NO_3)_2 \cdot 6H_2O$ was titrated with NaOH solution.
Sample 7.	A solution (7.0 L) of 0.3670 g of $Na_2SiO_3 \cdot 9H_2O$, 0.2826 g of $Fe(NO_3)_3 \cdot 9H_2O$, 0.4821 g of $Al(NO_3)_3 \cdot H_2O$, and 0.1680 g of $Zn(NO_3)_2 \cdot 6H_2O$ was titrated with Na_2CO_3 solution.
Sample 8911.	A solution (150 L) of 2.17 g of $Fe(NO_3)_3 \cdot 9H_2O$ was titrated with NaOH solution.
Sample 8912.	A solution of 34.7368 g of $Fe(NO_3)_3 \cdot 9H_2O$ was diluted to 150 L.
Sample 8913.	A solution of D-tank water was pH adjusted with NaOH.
Sample 8914.	A solution of 6.6 g of $Fe(NO_3)_3 \cdot 9H_2O$ was diluted to 150 L.
Sample 8915-1.	A solution of D-tank water was pH adjusted with NaOH.
Sample 8915-3.	A solution of D-tank water after recirculating through a pump for 23 hours, was pH adjusted with NaOH.
Sample 8916.	A solution of D-tank water was pH adjusted with Na_2CO_3.
Sample 8917.	A solution of 6.6 g of $Fe(NO_3)_3 \cdot 9H_2O$ and 8.1 g of $Na_2SiO_3 \cdot 9H_2O$ was diluted to 150 L.
Sample 8918.	A solution of 6.6 g of $Fe(NO_3)_3 \cdot 9H_2O$, 6.6 g of $Na_2SiO_3 \cdot 9H_2O$, and 6.0 g of $Al(NO_3)_3 \cdot H_2O$ was diluted to 150 L.

After the solutions were prepared, TS, TSS, TDS, and SDI measurements were conducted. The conductivity and pH of each solution was also determined. The results of these experiments are given in Table 2. Figures 1 - 3 contain data collected by J. Siler and are presented here with permission. The filtration unit used was a 2.2-sq ft Alcoa ceramic filter. The filter is constructed of alpha-alumina and has a pore size rating of 0.2 micron. The feed solution was prepared in a single batch (150 L) and recirculated. The vertical axis on Figures 1 - 3 is Jw/Jwo, which is the ratio of flux at any time to the initial flux.

RESULTS

When samples of deionized water with 2.0, 5.0, and 10.0 ppm of Fe (samples 1, 2, and 3 respectively) were run, the SDI values were calculated as being 1.23, 4.13, and 4.51 respectively. The results were as expected; the rate of fouling as well as the total time necessary to run the SDI increases with increasing solids content. When running solutions of filtered process water containing 2.0, 5.7, and 30.0 ppm of Fe (samples 8911, 8912, and 8914), the SDI values were determined to be 4.29, 2.72, and 1.95 respectively. However, the total time necessary for the SDI filtration increased significantly from 30 minutes to 100 minutes. The reason for this apparent reversal in SDI is attributed here to differences in precipitation characteristics. These differences are likely due to either the origin of the water or the method of preparation. The process water contains silica, clay, and dissolved salts which are not present in deionized water. The gelatinous nature and particle size of the precipitate can be affected by these species. The method of preparation differed slightly for the deionized water samples compared to the filtered process water samples. The deionized water samples began at pH = 1.0 during titration, but the filtered process water samples were prepared by addition of a pH = 1.0 solution containing the metals, to 150 L of approximately neutral water. This subtle difference in initial conditions may affect particle characteristics.

Samples 4, 5, and 6 were prepared in deionized water. All of these simulants contain Fe, but Si, Al, and Zn were added to determine their contribution to filter performance. The SDI and TSS data are presented in

Table 2. It can be seen that the SDI of the Fe and Si simulant (#4) is higher than that for simulants containing Al and Zn. The total filtration time for the Fe and Si simulant is less than that for those containing Al and Zn.

Figures 1-3 contain information collected on actual ceramic filters. This data was collected by J. Siler and is presented here with the author's permission. The filter performance curves are arranged by the type of simulant prepared. The solutions used for runs 8911, 8912, and 8914 are presented in Figure 1. These solutions were all prepared by the addition of iron(III) nitrate to process water. Figure 2 contains filter performance curves for runs 8913, 8915, and 8916. The simulant used for these runs is well-water containing small amounts of clay and iron(III) nitrate and is termed "D-tank". Figure 3 presents filter performance curves for 8914, 8917, and 8918. These simulants were prepared from filtered process water with added amounts of iron (III) nitrate, sodium metasilicate, and aluminum nitrate. Table 1 summarizes the preparation of all the simulant solutions.

Comparison of the TSS and SDI data for run 8911, 8912, and 8914 (2.0, 5.7, and 30 ppm Fe respectively) shows that as the TSS value increased, the SDI decreased. The TSS of both 8911 and 8914 turned out to be 26 ppm, but 8912 was 62 ppm. The SDI of samples 8911, 8912, and 8914 decrease with increased Fe content. The total time necessary for filtration increases significantly with increased Fe content. What this appears to suggest is that the rate of fouling decreases with increased solids, but the overall flux may be much lower with higher solids content.

Samples 8913, 8915, and 8916 were prepared from D-tank simulant treated in slightly different ways. Sample 8913 was simply pH adjusted to 7.5 pH units with sodium hydroxide (initial pH = 3.3). Sample 8915 was first treated with nitric acid (to pH = 1.0), then adjusted to pH = 7.5 with NaOH. Sample 8916 was similar to 8915, but was adjusted to pH = 7.5 with sodium carbonate. Sample 8915-1 was collected prior to passing through a centrifugal pump, and 8915-3 was collected after recirculating through a centrifugal pump for 23 hours. The TSS data for all four samples are over 200 mg/L. The SDI values obtained decrease in the order 8916 > 8913 > 8915. The time necessary for filtration decreases in the same order. It is interesting that the SDI and filtration times increase for 8915-3 compared to 8915-1. This tends to suggest that the recirculation through a centrifugal pump tears up the particles, and makes them more difficult to filter.

Samples 8914, 8917, and 8918 were prepared from filtered process water with added Fe (8914), Fe and Si (8917), and Fe, Si, and Al (8918). The TSS of these samples increased with the addition of Fe, Si, and Al as expected. The SDI of these samples is lowest for the Fe only simulant (8914), and highest for the Fe, Si, and Al simulant (8918). The total filtration time is lowest for the Fe only simulant, and highest for the Fe and Si simulant. This tends to suggest that the nature of the precipitate influences the filtration characteristics. A change in the precipitate composition such as a mixture of Fe and Si as compared to Fe only appears to change it's filterability. This result is the same as that for comparison of samples 2, 4, and 5, which were prepared from deionized water and vary slightly in concentration.

Runs 8 and 9 were used as a baseline for filtered process water. In run 8, the process water was tested directly from the source. In run 9, the water was pH adjusted to pH = 7.5 prior to testing. It was seen that although the SDI values are high, 5.9 and 6.0 for runs 8 and 9, the filtration time is very short. The contribution of solids in the process water does not appear to be significant without the addition of other reagents.

Figures 1-3 show that the ceramic filter performance is significantly affected by the method of preparation of the simulants. It was observed in several runs that sudden flux loss occurs at the beginning of an experiment. The flux decreases to some level in the first hour of operation and then declines more gradually, as in Figure 2.

The SDI of runs 8911, 8914, and 8912 would have predicted 8911(2 mg/L Fe) to foul the filter more rapidly than 8912(30 mg/L). As can be seen from Figure 1, this is reversed. The total time required to filter the solution in the SDI experiment increased from run 8911 to 8914, to 8912. This observation suggests that the total filtration time must be considered when estimating filter performance, and that higher feed solids may be beneficial to filter performance.

The SDI of runs 8913, 8915, and 8916 predict that filter performance would decrease in the order 8915 > 8916 > 8913. This is supported by the data in Figure 2. The total filtration time during the SDI experiment also predicts filter performance to decrease in the order 8915 > 8916 ≅ 8913.

The filter performance curves for simulants prepared with Fe, Al, and Si are presented in Figure 3. The SDI values would have predicted that the ordering would be 8914(Fe) > 8917(Fe + Si) > 8918(Fe + Si + Al). Total filtration time during the SDI experiment would have predicted 8914 > 8918 > 8917. The filter performance curves from Figure 3 are also in the order 8914 > 8917 ≅ 8918.

SUMMARY AND CONCLUSIONS

Analysis of the data that were collected during experimentation suggests that the relationship between SDI and filter performance is complex. The data suggest that both the total time (Ti + Tf) and the SDI must be considered when attempting to predict filter performance. Other parameters such as compressibility and rigidity of the precipitate can effect a difference in performance between SDI measurement and ceramic filter flux. The SDI unit employs a dead-end filtration and the ceramic filters are arranged in crossflow design. The affinity of the precipitate for adsorption onto the alumina membrane filter also cannot be accounted for here. Seeing that this is just a preliminary investigation, further tests must be completed. Varying the amounts of aluminum and silica is expected to affect the filterability of the simulant[4]. The method of precipitation, including the rate, concentration, and temperature are also expected to vary the filtration performance.

It is seen from this study that a comparison of the SDI data must be limited to simulants that contain similar compounds, These comparisons are useful only for relative information and should not be used for absolute prediction of filter performance. It is crucial that confirmatory tests using a ceramic filter be completed to prove the effects of various pretreatment chemistry. The SDI measurements can be used only as a preliminary screening tool to indicate expected filter performance.

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4. Jackson, J.M., Landolt, D., *Desalination*, 12, 361 (1973).

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Table 1. Estimates of Concentrations of Species (mg/L)

Exp't	Water	Fe	Si	Al	Zn	pH	rgt
1	0.1 M HNO_3	2.0	-	-	-	7.5	NaOH
2	0.1 M HNO_3	5.0	-	-	-	7.5	NaOH
3	0.1 M HNO_3	10.0	-	-	-	7.5	NaOH
4	0.1 M HNO_3	5.7	5	-	-	7.5	NaOH
5	0.1 M HNO_3	5.7	5	5	-	7.5	NaOH
6	0.1 M HNO_3	5.7	5	5	5	7.5	NaOH
7	0.1 M HNO_3	5.7	5	5	5	7.5	Na_2CO_3
8	f.p.w.	-	-	-	-	n.a.	n.a.
9	f.p.w.	-	-	-	-	7.5*	NaOH
8911	0.1 M HNO_3	2	-	-	-	7.5	NaOH
8912	f.p.w.	30	-	-	-	7.5	NaOH
8913	D-tank	-	-	-	-	7.5	NaOH
8914	f.p.w.	5.7	-	-	-	7.5	NaOH
8915-1	D-tank	-	-	-	-	7.5	NaOH
8915-3	D-tank**	-	-	-	-	7.5	NaOH
8916	D-tank	-	-	-	-	7.5	Na_2CO_3
8917	f.p.w.	5.7	5	-	-	7.5	NaOH
8918	f.p.w.	5.7	5	2.7	-	7.5	NaOH

*after stirring overnight at pH = 7.5
**after passing through pump

Table 2

Sample	pH	Conductivity (umho/cm)	TS (mg/L)	TSS (mg/L)	TDS (mg/L)	SDI	Ti (min:sec)	Tf (hr:min:sec)
1	7.25	18,600	32,281	36	31,888	1.23	9:12	11:47
2	7.07	6600	19,560	29	19.382	4.13	6:19	16:30
3	7.22	6400	18,865	39	18,914	4.51	10:09	31:24
4	7.33	11,400	11,686	37	10643	4.86	6:03	22:23
5	7.30	12,000	9909	56	9594	3.12	17:24	32:40
6	7.69	12,200	7825	43	7622	3.19	20:09	38:55
7	7.35	15,000	10,516	75	12,559	3.64	15:45	34:05
8	6.15	66	13,812	5	13,832	5.11	0:26	1:11
9	7.60	150	13,926	5	13,939	6.00	0:43	4:33

Table 2 (continued)

Sample	pH	Conductivity (umho/cm)	TS (mg/L)	TSS (mg/L)	TDS (mg/L)	SDI	Ti (min:sec)	Tf (hr:min:sec)
8911	9.00	2500	313	26	390	4.29	10:05	28:21
8912	9.38	495	424	62	559	1.95	41:03	58:05
8913	7.26	5200	4581	273	4222	2.77	45:22	55:48
8914	9.80	295	230	26	339	2.72	16:18	27:36
8915-1	6.30	14,400	12,510	321	11,044	1.47	17:49	22:44
8915-3	6.75	15,200	12,401	210	12,043	5.24	8:47	39:47
8916	7.80	14,000	13,272	501	12,202	4.66	22:14	1:13:43
8917	9.04	260	361	36	234	4.17	47:51	2:07:22
8918	8.28	320	550	42	561	4.58	23:26	1:14:17

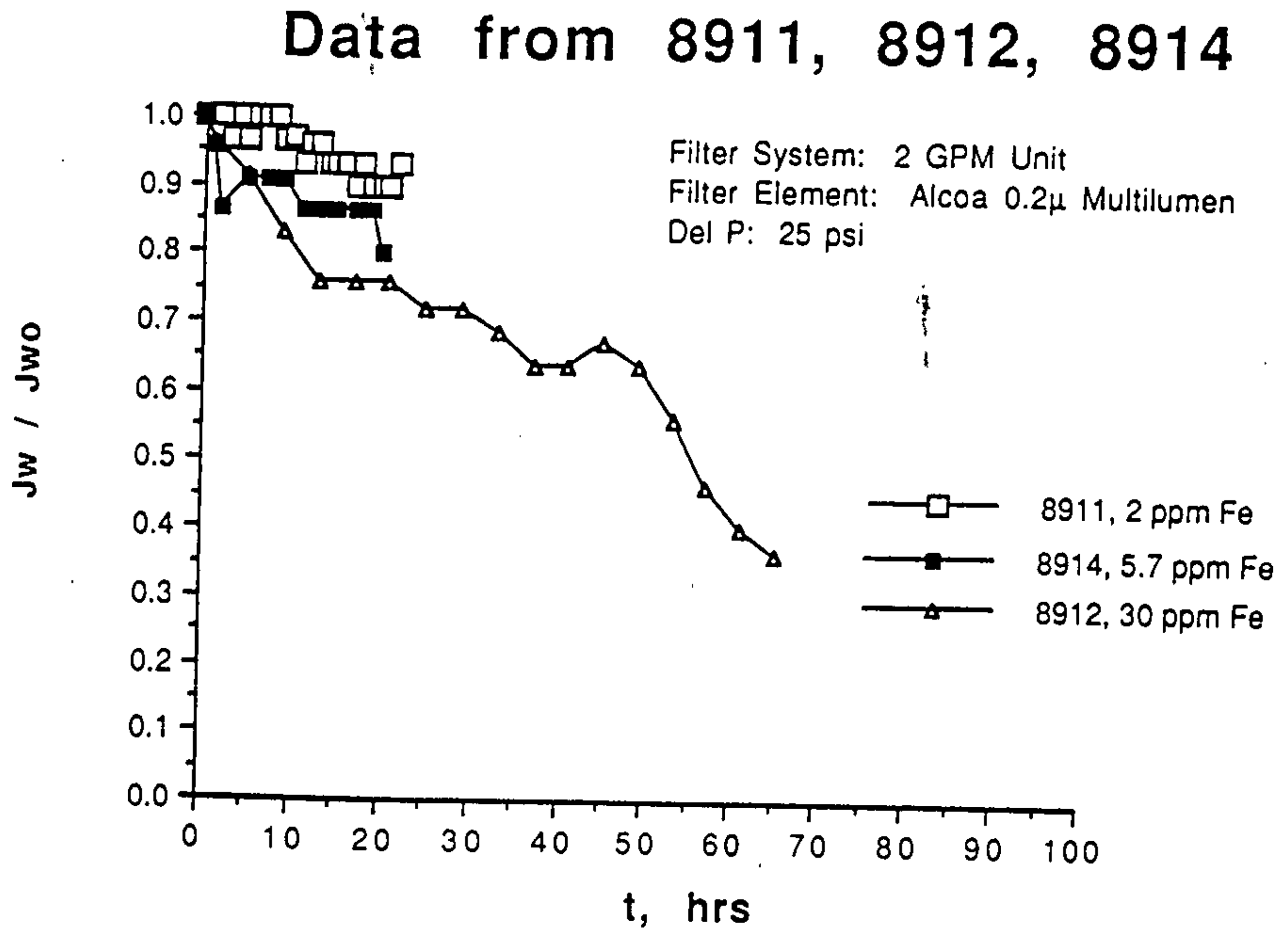


Figure 1. Filtered Process Water with Various Amounts of Fe.

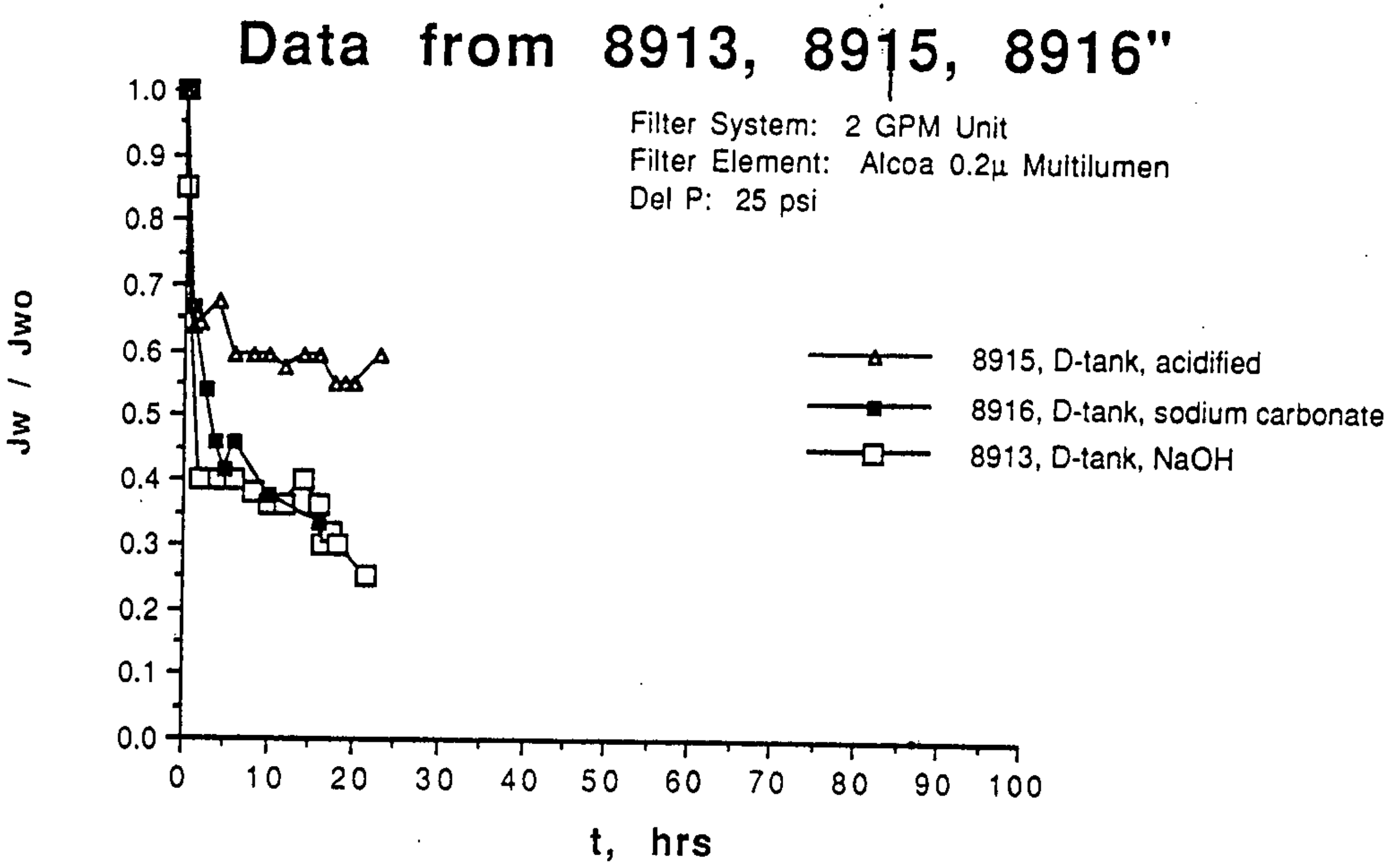


Figure 2. D-Tank Water

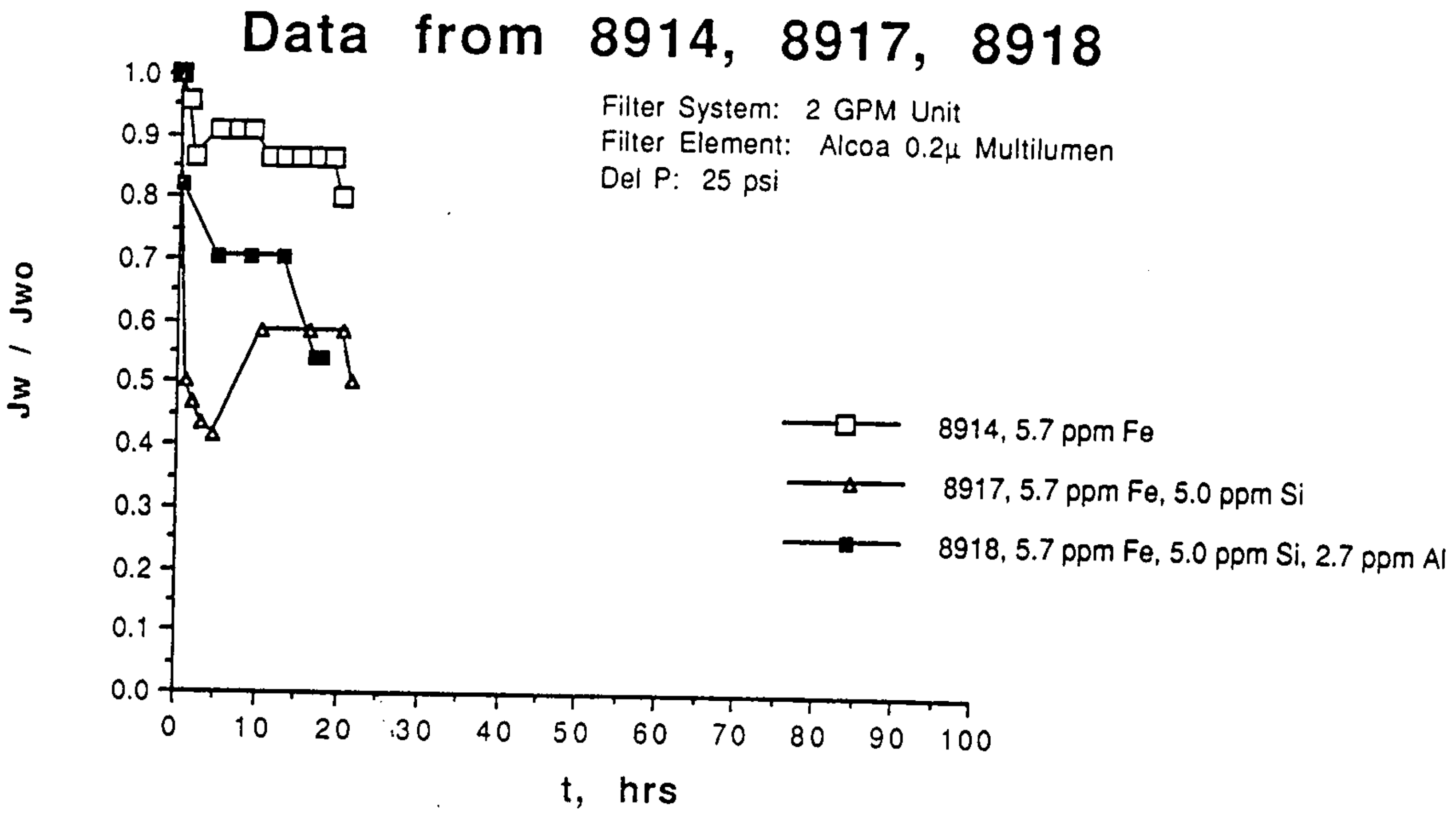
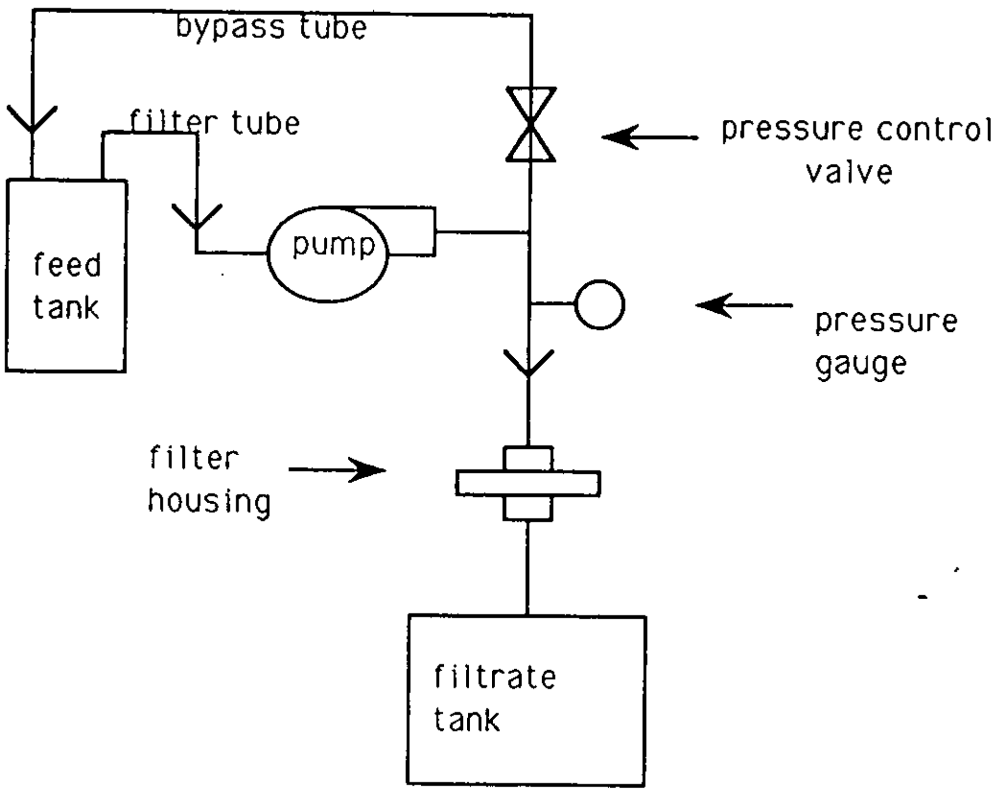


Figure 3. F.P.W. with Various Amounts of Fe, Si, and Al.



Schematic 1. Silt Density Index Apparatus

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