

Chapter 3

Experimental Program

3.1 Sample Preparation

Past studies have identified urban stormwater runoff as a major contributor to the degradation of many urban lakes, streams, and rivers. Industrial and commercial parking lots, material storage areas, and automobile service stations are the most significant contributors of a variety of pollutants to wet weather flow. Therefore, an attempt was made to analyze both stormwater runoff and synthetic samples. The former was intended to provide a preliminary guideline in parking lot runoff characteristics, while the latter allowed a consistent background for experimentation. Preparation and evaluation procedures for each type of sample are described in this section. In addition, CSO samples were also used for this study.

Surface Runoff

During the initial stage of the investigation, surface runoff samples were collected from a parking lot. In addition to natural storm surface runoff samples, simulated runoff was generated by spraying the above-noted parking lot with city water during dry weather periods.

The parking lot used in this study was the Otto H. York Center Parking Lot #3 at the New Jersey Institute of Technology (NJIT) campus in Newark, New Jersey. The parking lot has a capacity of 60 cars with a dimension of approximately 100 meters by 30 meters with a 4% grade. It operates seven days per week with five days at full capacity.

Sampling of actual stormwater runoff events is weather dependent, and hydrologic factors such as the interval between storm events, rainfall intensity, total rainfall, etc. are random in nature, and, as such, greatly affect runoff characteristics. Because of this, the time to conduct the experiments and the ability to replicate results with varying samples would have required a substantially longer period than is programmed in the investigation. Therefore, actual runoff samples were used only in the preliminary stage of this study to test the feasibility of the MC process.

Sampling.

Bulk runoff samples generated by natural rain or by spraying a predetermined area of the parking lot with city water were sampled at the beginning of the precipitation event directly from the drainage inlet chamber by a hand-bucket and industrial vacuum apparatus. The pavement was washed from the highest elevation to the lowest elevation. The total area washed for each of the

tests was approximately 3,000 square feet. Sampling setup and operations were prepared at least two days prior to rainfall events anticipated in accordance with the weather forecast.

Sample Preservation, Transportation, and Storage.

Samples were transported to the laboratory within five minutes after finishing the sample collection process. The preservation and holding time of samples are listed in Table 3-1.

Table 3-1. Preservation Condition and Holding Time For Sample Analysis

Parameter	Container	Preservation	Holding Time
Particle size distribution	P, G*	Cool, 4°C	48 hours**
Zeta potential	P, G	Cool, 4°C	48 hours**
Suspended solid	P, G	Cool, 4°C	7 days
Total solid	P, G	Cool, 4°C	7 days
Volatile solids	P, G	Cool, 4°C	7 days
pH	P, G	None	Immediately
Fecal coliform	P, G	Cool, 4°C	6 hours
Total organic carbon	P, G	Cool, 4°C	28 days
Turbidity	P, G	Cool, 4°C	48 hours

* P -- plastic container; G -- glass bottle

** No standard holding time is given in the currently available literature. This number is selected based on turbidity holding time.

Synthetic Samples.

As indicated above, evaluation of the MC process by both natural and simulated runoff requires a substantial amount of sampling due to the randomness of runoff samples. Since the dry residual materials from a parking facility also possess the basic constituents of parking lot runoff, such residues were collected

and used to prepare synthetic samples. It was found that two types of dry materials existed:

Type I -- Low organic content materials. The type I material is a low organic content sandy-like residue that was found in the area along the walls of the parking deck. The total volatile solid content of this type of material was found to be less than 1% by weight. This material was sieved and divided into the seven particle size ranges as shown in Table 3-2.

A series of sample preparation evaluation tests were conducted to determine the quantity of each range for the aqueous sample to be analyzed. It was found that the material with grain size larger than 106 μm would settle too fast to make a uniform sample. It was concluded that particles smaller than 106 μm were more suitable for suspension than the larger particles. Subsequently, particles smaller than 106 μm were used to prepare synthetic samples. The constituents of the synthetic samples are illustrated in Table 3-3.

Table 3-2. Dry Sample Size Characteristics

Particle Diameter (μm)	Control Sieve No. (ASTM)
less than 53	270
53 — 75	270 — 200
75 — 106	200 — 140
106 — 150	140 — 100
150 — 250	100 — 60
250 — 425	60 — 40
larger than 425	40

Table 3-3. Composition of Synthetic Samples

Material Type	Specification	Relative Weight*	Remarks
Type I Low organic content material	size < 53 (μm)	38.9%	By sieve analysis
	53 < size < 75 (μm)	19.4%	
	75 < size < 106 (μm)	8.3%	
Type II with Clay	High organic content	1.9%	High organic content materials were ground with clay
	0.1 < size < 3 (μm)	31.4%	
Total		100%	

* based on the total weight of dry sample

Type II -- High organic content material. The type II material is a high organic content residue that was attached to the ground surface under each parked car. The total volatile solids for this type of material were found to be approximately 18% by weight. In performing sample preparation tests, it was observed that this material either settles at the bottom of the jar or floats on the surface. In order to obtain suspended volatile solids, the high organic content material was first ground with clay and then used to prepare an aqueous sample. After grinding with clay, it was observed that the type II solids remained in a suspended state, which was confirmed by total volatile solid testing. A series of tests were conducted to determine the proportion between clay and the original sample. Both low and high organic content materials were combined and used to generate a synthetic sample. The composition of the type II material ground with clay is shown in Table 3-3.

Combined Sewer Overflow

The City of Perth Amboy operates a combined sewer system and wastewater transfer pumping station that collects combined sanitary sewage, industrial wastewater, and storm runoff from an approximately 7 square kilometers drainage area to a regional wastewater treatment plant owned and operated by the Middlesex County Utilities Authority. The wastewater transfer pumping facility is located at the junction of Water Street and Sadowski Parkway. A CSO regulator is directly located about 20 feet below the Sadowski Parkway with an overflow weir and 84" diameter CSO tide-gated outfall to the Arthur Kill.

The inflow from the interceptor discharges into one of two screening channels each equipped with a mechanics coarse bar screen for removing screenings and protecting the sewage pumps. The screen chamber inflow was collected for the MC coagulation study.

The channel is housed in an enclosed building with 24 hours a day access and a person always on duty. Grit, that accumulates in the channel, is removed from the facility monthly.

3.2 Measurement Parameters

In this experimental program, the parameters were divided into control and response variables.

Control Variables

Control variables are independent variables of a system whereas response variables are dependent variables (results). In this study, the control variables included the following parameters:

- MC type, size range, and concentration.
- Coagulant type and concentration.
- Coagulant aid (coagulant aid) type and concentration.
- Rapid mixing time and rotation rate.
- Slow mixing (flocculation) time and rotation rate.

Response Variables

Response variables are indicators used for determining the effectiveness of the control variables. Response variables may provide useful information in determining the optimal setup for control variables. In this study, the following parameters were used as response variables:

- Supernatant turbidity.
- Supernatant pH.
- Supernatant particle-size-distributions.
- Supernatant zeta potential.
- Supernatant suspended solids.
- Supernatant total solids.
- Supernatant volatile solids.
- Supernatant fecal coliform.
- Supernatant total organic carbon.

Raw Stormwater Characterization. The raw samples including surface runoff, synthetic samples, and CSO (see Section 3.1) were characterized by the following parameters:

pH	Total volatile solids
Turbidity	Zeta potential
Total solids	Total organic carbon
Particle size distribution	Fecal coliform
Suspended solids	

3.3 Experimental Design

The experimental design of this study consisted of a three-layer experimental design: prescreening tests, screening tests, and confirmative tests. The purpose of the prescreening tests during which different experimental conditions were evaluated by visual observation was to determine the range of operational parameters. Upon the completion of the prescreening tests, screening tests were performed followed by confirmative tests. The purpose of screening and confirmative tests was to provide a quantitative analysis of the MC process. Detailed descriptions of MC, coagulant, coagulant aid, prescreening tests, screening tests, and confirmative tests are given in the following sections.

Microcarrier (MC)

Two types of material were selected to use as MC, namely, Ottawa sand and a beach sand obtained from Sandy Hook, NJ. Both sands were used in prescreening tests. Ottawa sand was selected in screening and confirmative testing, due to its durability and size uniformity over the Sandy Hook beach sand and commercial available in large quantity. The size range of Ottawa sand tested was between 100 to 500 μm . The MC size ranges and concentrations were determined by prescreening tests.

All containers and MC were first washed thoroughly with a detergent and hot water, then rinsed with hot water to remove all traces of residual washing compound, and finally rinsed with particle-free water.

Coagulant

Alum (aluminum sulfate, $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$) and ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) were used as the coagulants for this study. Aluminum sulfate has been employed extensively in water and wastewater treatment because it is usually less expensive than other coagulants and it operates effectively close to neutral pH's while ferric chloride is effective over a wider pH range. Concentrations of coagulant reported in this report are the concentration of aluminum sulfate as mg of $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}/\text{L}$ and the concentration of ferric chloride as mg of Fe^{+++}/L .

The zeta potential and pH of aluminum sulfate and ferric chloride were measured at different concentrations. Stock coagulant test solutions were prepared daily by mixing chemicals with deionized water to a concentration of 10 g/L (1 mL of stock solution when added to 1 L of sample is equivalent to 10 mg/L). The zeta potential and pH values of the deionized water were close to 0 mV and 7, respectively.

The zeta potential distributions of aluminum sulfate and ferric chloride solutions for different concentrations are presented in Figures 3-1 and 3-2. The zeta potential increases with the increase of concentration within the range of 10—100 mg/L for both coagulants. For concentrations higher than 100 mg/L, no significant changes for zeta potential was noticed.

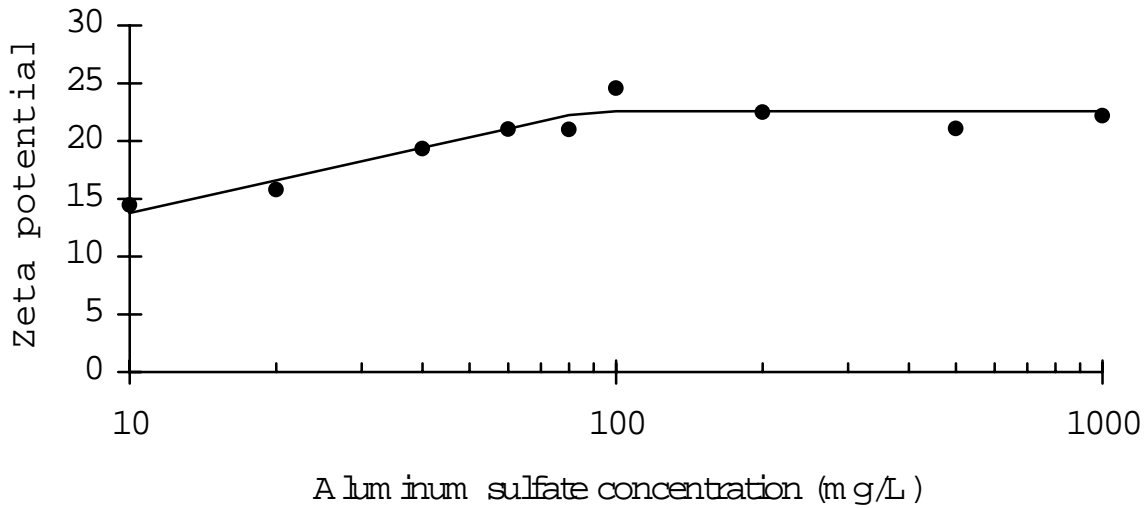


Figure 3-1. Zeta Potential Distribution of Aluminum Sulfate Solution

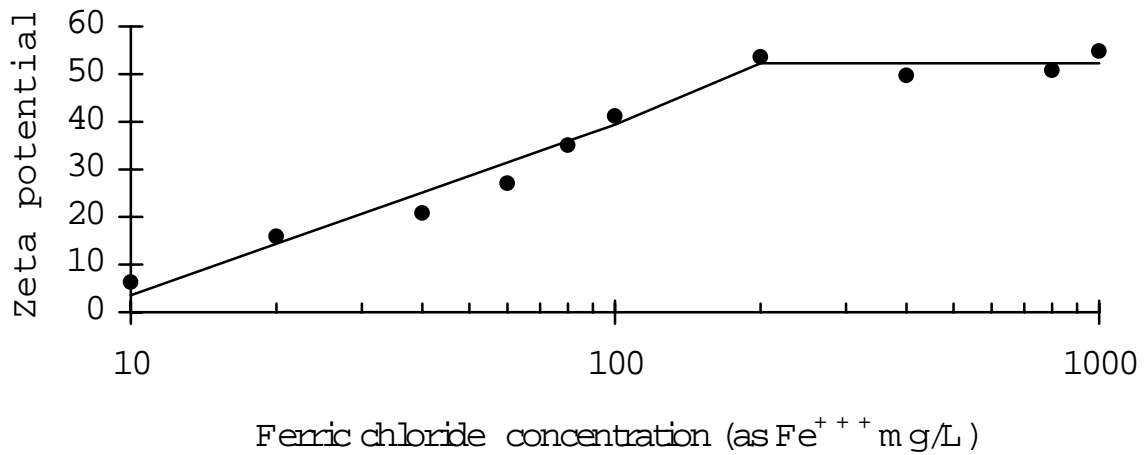


Figure 3-2. Zeta Potential Distribution of Ferric Chloride Solution

Figures 3-3 and 3-4 illustrate variation of pH values with different concentrations of coagulant. The pH value decreases from 5.2 to 3.5 for aluminum sulfate concentrations increases from 10 to 1000 mg/L as $Al_2(SO_4)_3 \cdot 18H_2O$, and the pH values vary from 3.5 to 2.3 for ferric chloride solution when the concentration of ferric chloride increases from 10 to 1000 mg/L as Fe^{+++} (or 48 to 4,800 mg/L as $FeCl_3 \cdot 6H_2O$).

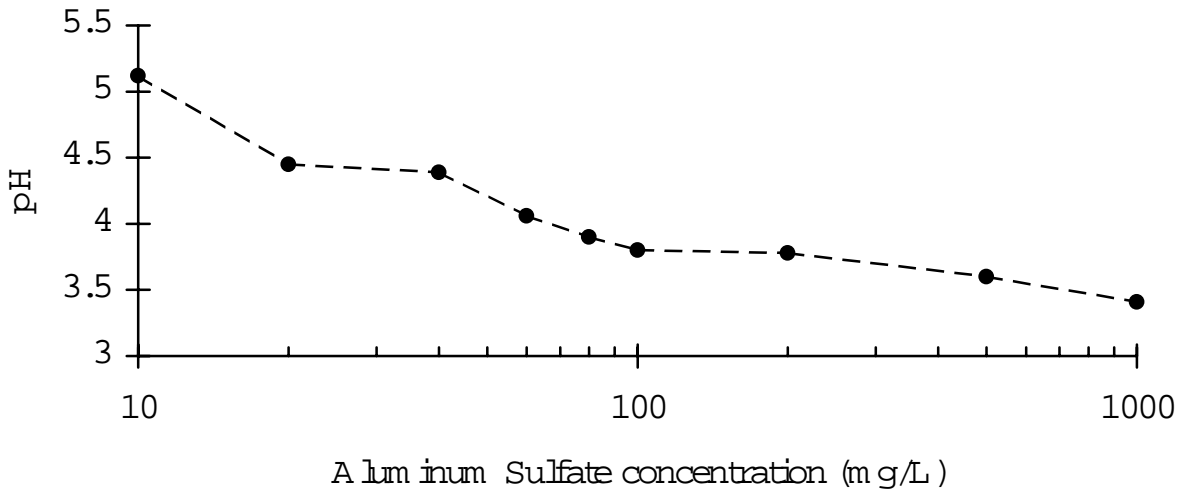


Figure 3-3. pH Distribution of Aluminum Sulfate Solution

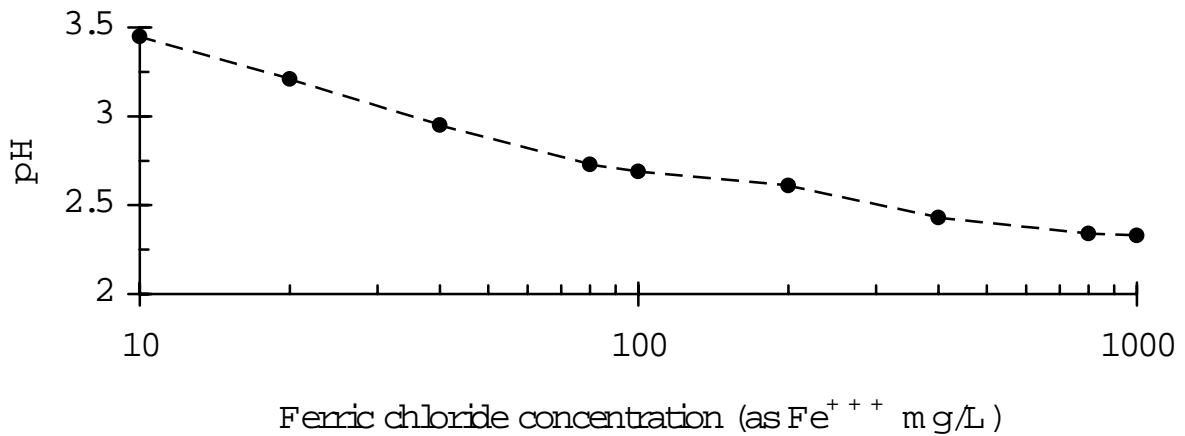


Figure 3-4. pH Distribution of Ferric Chloride Solution

Coagulant Aid

There are many commercially available coagulant aids or polyelectrolytes. Five polyelectrolytes from two different manufacturers (see Table 3-4) were used in the experiments. Among them, four (POL-EZ-2466, POL-EZ-3466, POL-EZ-2696, and POL-EZ-7736) were used in surface runoff tests and one (309C) for CSO tests. Zeta potential distributions versus polyelectrolytes concentrations are illustrated in Figures 3-5, 3-6, 3-7, and 3-8, respectively. For cationic and anionic polyelectrolytes, the charge strengths are stronger for higher concentrations, while for non-ionic polymer, there is no significant change in zeta potential when concentration increases.

The relationship between coagulant aid concentrations and zeta potential values based on log-linear regression is summarized in Table 3-5 and illustrated in Figure 3-9. One can see that the cationic (POL-EZ-2466 and POL-EZ-3466) and anionic (POL-EZ-7736) coagulant aids have similar strengths of charge while the zeta potential of the non-ionic coagulant aid (POL-EZ-2696) is comparatively insignificant.

Table 3-4. List of Coagulant Aids

Coagulant Aid (Polyelectrolyte)	ID	Charge	Test Stage	Manufacturer
POL-EZ-2466	PE-1	Cationic	Surface Runoff	Calgon Corporation Pittsburgh Pennsylvania
POL-EZ-3466	PE-2	Cationic		
POL-EZ-2696	PE-3	Non-ionic		
POL-EZ-7736	PE-4	Anionic		
309C	PE-5	Cationic	CSO	Polydyne, Inc Riceboro Georgia

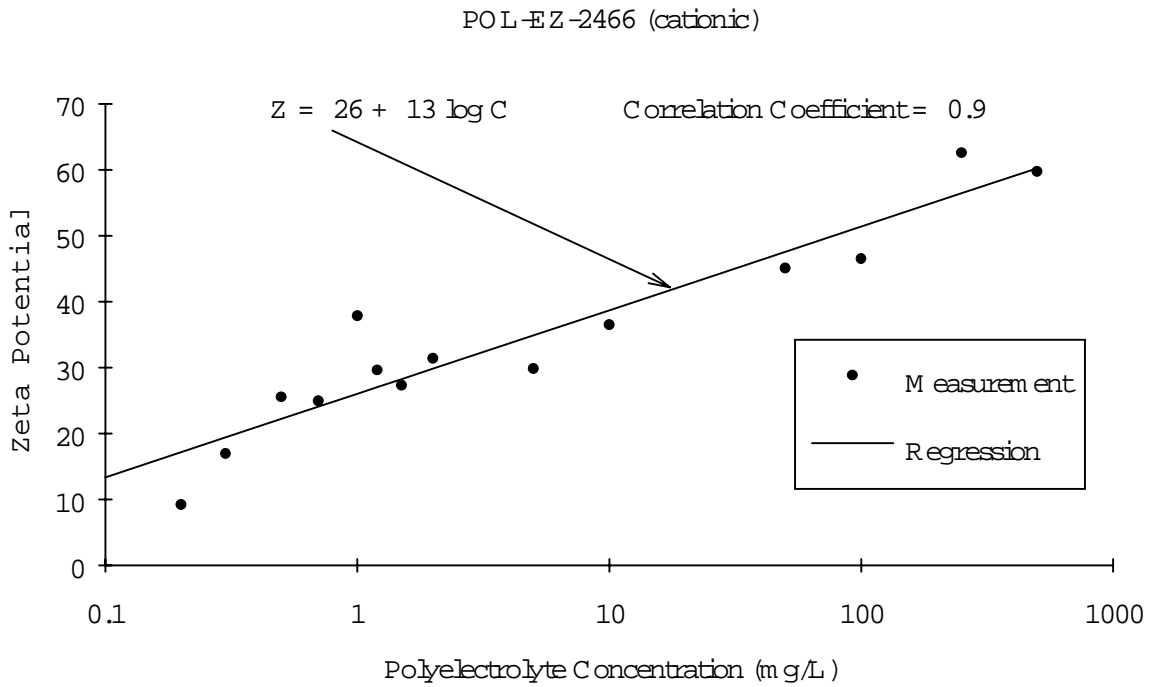


Figure 3-5. Zeta Potential Distribution of POL-EZ-2466

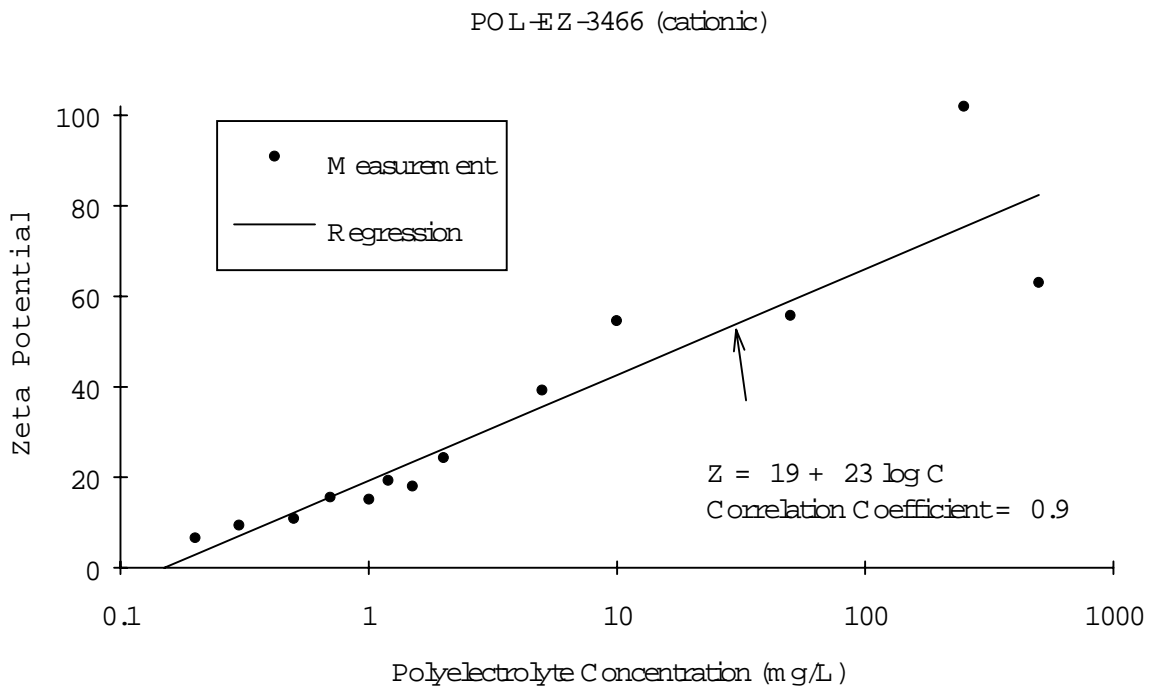


Figure 3-6. Zeta Potential Distribution of POL-EZ-3466

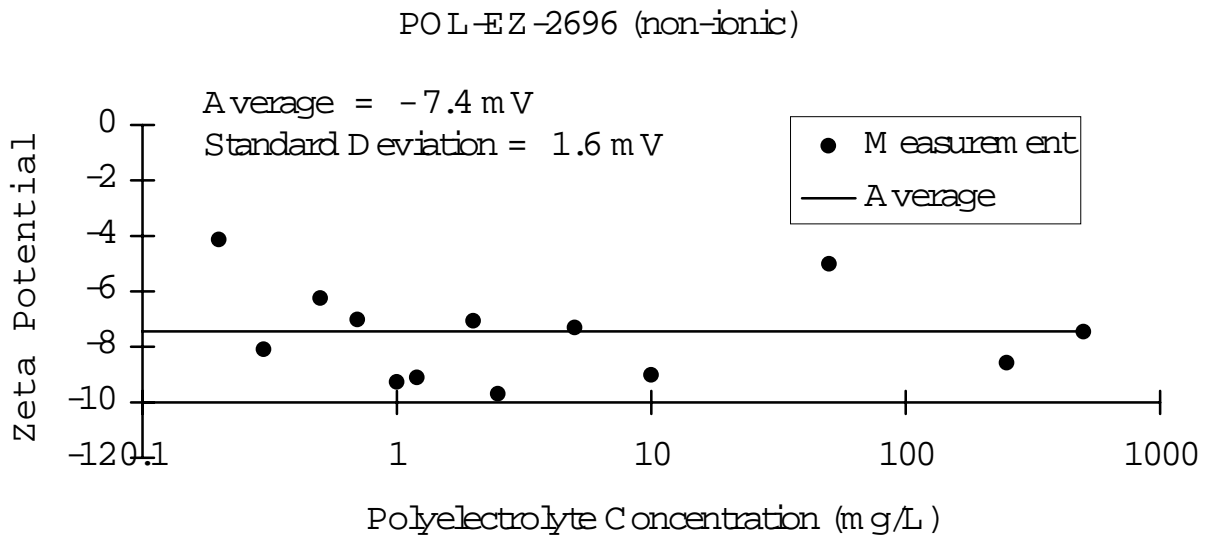


Figure 3-7. Zeta Potential Distribution of POL-EZ-2696

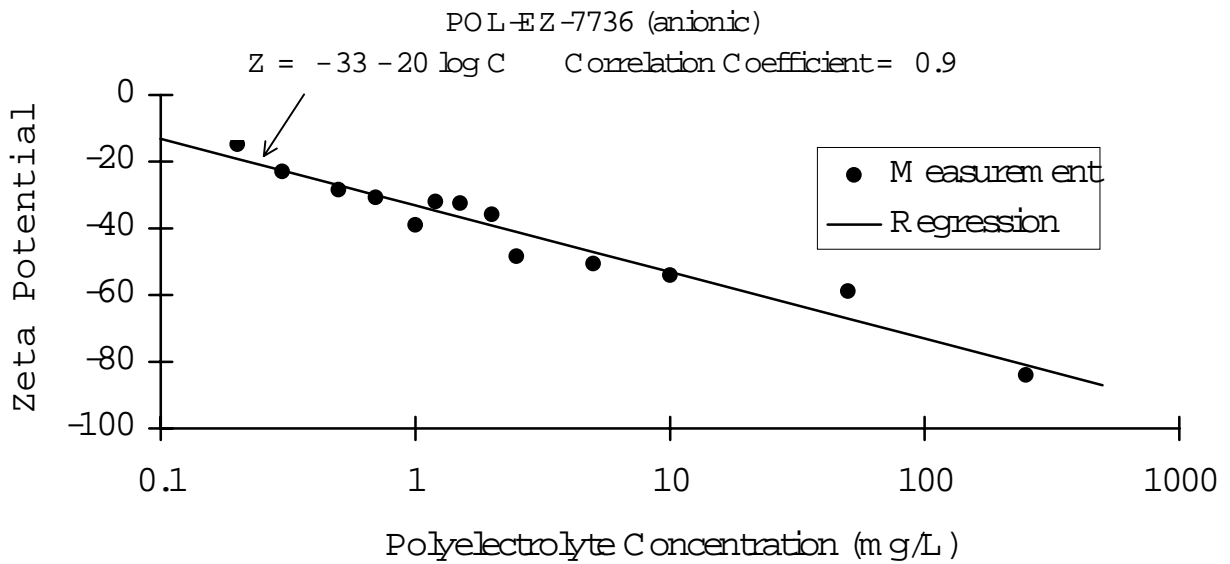


Figure 3-8. Zeta Potential Distribution of POL-EZ-7736

Table 3-5. Zeta Potential of Coagulant Aids

Coagulant aid	Zeta Potential versus Concentration
POL-EZ-2466	$Z = 26 + 13 \log C$
POL-EZ-3466	$Z = 19 + 23 \log C$
POL-EZ-2696	$Z_{\text{average}} = -7.4$ Standard deviation = 1.6 mV
POL-EZ-7736	$Z = -33 - 20 \log C$

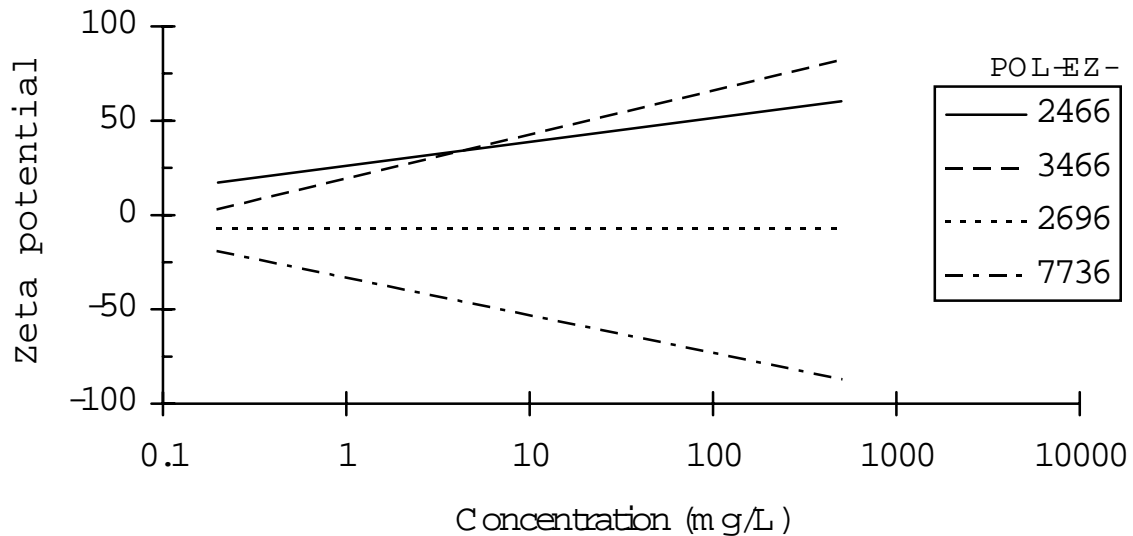


Figure 3-9. A Comparison of Zeta Potential of Coagulant Aid

Experiments

The experimental program consisted of three phases, namely, prescreening, screening, and confirmative tests. The prescreening phase was a qualitative determination that provided operational setups for the screening and confirmative tests. The screening tests evaluated various coagulant and coagulant aid concentrations for the MC jar tests. Finally, the confirmative tests assessed the effects of MC size and concentration in the jar test. The function and program of each phase is described in detail in the following sections.

Prescreening Jar Tests

The prescreening tests consisted of a qualitative characterization to provide testing ranges for the control variables (see Section 3.2). In the prescreening tests, a series of jar tests was performed on natural and artificial storm runoff samples mixed with MC, coagulant and coagulant aid. The operational parameters included: coagulant aid type and dosage, coagulant dosage, rapid mixing rate and duration, flocculation mixing rate and duration, and MC type, concentration, and size. The degree of agitation, time required for good floc formation, and the time required for settling were noted. Since the results are largely comparative, visual observation was employed. Quantitative analysis was considered unnecessary. The parameters and their ranges are listed in Table 3-6.

Table 3-6. Parameter Setup for Prescreening Jar Tests

Parameter	Material	Value Ranges
MC Size	Ottawa sand Beach sand	100—500 μm
MC Concentrations	Ottawa sand Beach sand	3—10 g/L
Coagulant	Aluminum sulfate	10—120 mg/L
Coagulant aid	POL-E-Z 3466	0.3—1.5 mg/L
Coagulant aid	POL-E-Z 2466	0.3—1.5 mg/L
Coagulant aid	POL-E-Z 7736	0.3—1.5 mg/L
Coagulant aid	POL-E-Z 2696	0.3—1.5 mg/L
Rapid Mixing Rate	—————	60—200 rpm
Flocculation Rate	—————	10—60 rpm
Rapid Mixing Duration	—————	10—120 sec
Flocculation Duration	—————	10 sec—30 min
Settling Time	—————	1—30 min

The minimum energy and time that can initiate all the MC into suspension were selected as the rapid mixing rate and duration for the jar tests. In addition, the minimum rate to keep all MC in suspension was selected as the optimal flocculation rate. The optimal flocculation time was determined based on floc formation. The rapid mixing rate and duration as well as the flocculation rate and duration were determined by visual observation. A summary of parameters with respect to this criteria selection is shown in Table 3-7.

Five MC configurations with different sizes and concentrations were selected based on prescreening tests and they are identified in Table 3-8.

Table 3-7. Determination of Rapid Mixing Rate with Duration and Flocculation Rate with Duration

Parameter	Selection Criterion
Rapid mixing rate	Minimum energy to initiate MC into the suspension
Rapid mixing duration	Minimum time to achieve fully mixed condition
Flocculation rate	Minimum energy to keep all MC and floc in the suspension
Flocculation duration	Best floc formation (size and density) at observation intervals of 1, 3, 5, 10, 15, 20, and 30 min

Screening Tests

The screening tests focused on determining the optimal doses of coagulant and coagulant aid for the MC process. There were three test levels with different combinations of MC, coagulant and coagulant aid. A summary of parameter evaluation for screening as well as confirmative tests is presented in Table 3-9.

Table 3-8. MC Identification

MC type	Material	Size	Dosage (g/L)
MC-1	Ottawa sand	53—150 μm (Sieve #270—#100)	3
MC-2	Ottawa sand	150—250 μm (Sieve #100—#60)	3
MC-3	Ottawa sand	53—150 μm (Sieve #270—#100)	10
MC-4	Ottawa sand	150—250 μm (Sieve #100—#60)	10
MC-5	Ottawa sand	53—75 μm (Sieve #270—#200)	3

Table 3-9. Screening and Confirmative Tests Parameter Evaluation

Test	Parameter	Control Variable
Screen level 1	MC-1 and Coagulant	Coagulant concentration
Screen level 2	MC-1, Coagulant, and Coagulant aid	Coagulant concentration
Screen level 3	MC-1, Coagulant, and Coagulant aid	Coagulant aid concentration
Confirmative	MC-1, MC-2, MC-3, MC-4, Coagulant, and Coagulant aid	MC size MC concentration

Rate and duration for rapid mixing and flocculation determined in prescreening tests were used for the screening tests. Turbidity was used as a water quality indicator. Supernatant samples were taken at settling times of 3 and 8 minutes, respectively.

Determination of optimal dose was based on the following criteria:

- Select the jar with the lowest turbidity, unless
- The difference between the lowest turbidity jars is less than 20%, in which case, select the jar with the lower chemical dosage.

Level 1. The purpose of Level 1 testing was to determine the best coagulant dosage in the absence of coagulant aid. MC-I was used for each jar. Coagulant concentration setup, ranging from 0 to 80 mg/L, is illustrated in Table 3-10.

Table 3-10. Screening Tests -- Level 1

Test Set	Additive	Jar No.					
		1	2	3	4	5	6
1-1	MC	M C - 1					
	Coagulant (mg/L)	0	10	20	40	60	80

Level 2. The purpose of level 2 testing was to determine the best coagulant dosage in the presence of coagulant aid. The influence of coagulant aid on the optimal dosage of coagulant was thus determined. Coagulant, MC-I, and 1 mg/L coagulant aid were used in the tests. The coagulant concentration setup, ranging from 0 to 80 mg/L, is illustrated in Table 3-11.

Level 3. The purpose of level 3 testing was to determine the influence of coagulant aid concentrations. The optimal coagulant concentration based on the test results of level 2, and MC-I was used for each jar. The coagulant aid concentration setup, ranging from 0.3 to 1.5 mg/L, is presented in Table 3-12.

Table 3-11. Screening Tests -- Level 2

Test Set	Additive	Jar No.					
		1	2	3	4	5	6
2-1	MC	M C - 1					
	Coagulant (mg/L)	0	10	20	40	60	80
	Coagulant aid PE-1 (mg/L)	1	1	1	1	1	1
2-2	MC	M C - 1					
	Coagulant (mg/L)	0	10	20	40	60	80
	Coagulant aid PE-2 (mg/L)	1	1	1	1	1	1
2-3	MC	M C - 1					
	Coagulant (mg/L)	0	10	20	40	60	80
	Coagulant aid PE-3 (mg/L)	1	1	1	1	1	1
2-4	MC	M C - 1					
	Coagulant (mg/L)	0	10	20	40	60	80
	Coagulant aid PE-4 (mg/L)	1	1	1	1	1	1

Table 3-12. Screening Tests -- Level 3

Test Set	Additive	Jar No.					
		1	2	3	4	5	6
3-1	MC	M C - 1					
	Coagulant (mg/L)	based on results from test set 2-1					
	Coagulant aid PE-1 (mg/L)	1	0.3	0.5	0.7	1.2	1.5
3-2	MC	M C - 1					
	Coagulant (mg/L)	based on results from test set 2-2					
	Coagulant aid PE-2 (mg/L)	1	0.3	0.5	0.7	1.2	1.5
3-3	MC	M C - 1					
	Coagulant (mg/L)	based on results from test set 2-3					
	Coagulant aid PE-3 (mg/L)	1	0.3	0.5	0.7	1.2	1.5
3-4	MC	M C - 1					
	Coagulant (mg/L)	based on results from test set 2-4					
	Coagulant aid PE-4 (mg/L)	1	0.3	0.5	0.7	1.2	1.5

Confirmative Tests

In confirmative tests, it was intended to determine the impact of the MCs with respect to their size and concentration. In these tests, one coagulant, four MCs, and four coagulant aids were used. The experimental setup is outlined in Table 3-13.

Table 3-13. Confirmative Tests

Test Set	Additive	Jar No.				
		1	2	3	4	5
C-1	MC	M C - 1				
	Coagulant (mg/L) and Coagulant aid (mg/L)	Based on Test Set 1-1	Based on Test Set 3-1	Based on Test Set 3-2	Based on Test Set 3-3	Based on Test Set 3-4
C-2	MC	M C - 2				
	Coagulant (mg/L) and Coagulant aid (mg/L)	Based on Test Set 1-1	Based on Test Set 3-1	Based on Test Set 3-2	Based on Test Set 3-3	Based on Test Set 3-4
C-3	MC	M C - 3				
	Coagulant (mg/L) and Coagulant aid (mg/L)	Based on Test Set 1-1	Based on Test Set 3-1	Based on Test Set 3-2	Based on Test Set 3-3	Based on Test Set 3-4
C-4	MC	M C - 4				
	Coagulant (mg/L) and Coagulant aid (mg/L)	Based on Test Set 1-1	Based on Test Set 3-1	Based on Test Set 3-2	Based on Test Set 3-3	Based on Test Set 3-4