BRACKISH GROUNDWATER TREATMENT AND CONCENTRATE DISPOSAL FOR THE HOMESTEAD COLONIA EL PASO, TEXAS

Civil Engineering Department College of Engineering University of Texas at El Paso El Paso, Texas 79968

by

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List of Abbreviations

BDF	brine disposal facility
CA	cellulose acetate
CERM	Center for Environmental Resource Management
ED	electrodialysis
EDAP	Economically Distressed Areas Program
EEP	Enhanced Evaporation Pond
EPWU	El Paso Water Utilities
gpd	gallons per day
gpm	gallons per minute
HMUD	Homestead Municipal Utility District
kgal	kilogallon
MCL	maximum contaminant level
MG	million gallons
MGD	million gallons per day
MPa	mega Pascal
MSFD	multi-stage flash distillation
N F	nanofiltration
PDP	Pilot Desalination Plant
PI	Principal Investigator
O&M	operation and maintenance
QA	quality assurance
QC	quality control
RO	reverse osmosis
SDI	Silt Density Index
SGSP	salinity gradient solar pond
TDS	total dissolved solids
TFC	thin-film composites
TNRCC	Texas Natural Resource Conservation Commission
TWDB	Texas Water Development Board
USBR	United States Bureau of Reclamation
USGS	United States Geological Survey
UTEP	University of Texas at El Paso

Metric Conversion Table

Multiply	Вү	To obtain
Length		
Inches (in)	25.4 (exactly)	Millimeters (mm)
Inches (in)	2.54 (exactly)	Centimeters (cm)
Feet (ft)	30.48 (exactly)	Centimeters (cm)
Feet (ft)	0.3048 (exactly)	Meters (m)
Feet (ft)	0.0003048 (exactly)	Kilometers (Km)
Miles (mi)	1.609344 (exactly)	Kilometers (Km)
Area		
Square inches (sq.in.)	6.4516 (exactly)	Square Centimeters (cm ²)
Square feet (sq.ft.)	929.03	Square Centimeters (cm ²)
Square feet (sq.ft.)	0.092903	Square meters (m ²)
Acres (ac)	4,406.9	Square meters (m ²)
Volume		
Cubic inches (cu.in.)	16.3871	Cubic centimeters (cm ³)
Cubic feet (ft ³)	0.0283168	Cubic meters (m ³)
U.S. Gallons (gal)	3.78533	Liters (1)
US. Gallons (gal)	0.00378543	Cubic meters (m ³)
Acre-feet (ac-ft)	1,233.5	Cubic meters (m ³)
Acre-feet (ac-ft)	1,233,500	Liters (1)
Pressure		
Pound / square in (psi)	6891	pascal (Pa)
Flow		
Cubic feet per second (cfs)	0.0283 17	Cubic meters/second (m^3/s)
Cubic feet per minute (cfm)	0.4719	Liters/second (1/s)
U.S. Gallons per minute (gpm)	0.06309	Liters/second (l/s)
Work and Energy		
British thermal unit (BTU)	1,055.06	joules (J)
BTU per pound (BTU/lb)	2.326 (exactly)	joules/gram (J/g)
Foot-pounds (ft-lb)	1.35582	joules (J)
Power		
BTU per hour (BTU/hr)	745.700	watts (W)
Foot-pounds/second (ft-lb/s)	1.35582	watts (W)

1.0 Executive Summary

The supply of brackish groundwater in the El Paso del Norte region is much larger than the fresh groundwater supply. Hydrologists predict that the region's fresh groundwater in the Hueco Bolson (aquifer) will be exhausted by 2025 at current use rates. Since Texas limits public drinking water supplies to a total dissolved solids (TDS) content of 1000 mg/l, groundwater with a TDS concentration in excess of this limit can be defined as brackish and unsuitable for municipal use without desalination. This brackish groundwater is a valuable future water resource, as is true for many regions in the southwest.

Utilization of brackish groundwater is dependent on the application of membrane technology for desalination. The development of low-pressure thin-film composite membrane technology has made the treatment of brackish groundwater in the large volumes needed for municipal supply economically feasible.

The single largest problem with the desalination of brackish groundwater in land-locked regions, such as El Paso, is the disposal or utilization of the reject (concentrate). The concentrate cannot be discharged to streams and deep well injection has its own set of problems such as clogging of the formation, corrosion of the well casing, and ground water contamination. When land is inexpensive, as it is in many regions of the southwest, evaporation becomes a feasible option. One of the largest expenses associated with concentrate disposal by evaporation is the lining of evaporative ponds with an impermeable barrier to prevent contamination of the underlying groundwater.

One of the major purposes of this research was to determine the feasibility of using salts contained in the concentrate to form a self-sealing barrier in evaporation ponds. This was done while providing the Homestead Municipal Utility District (HMUD) with 50,000 gpd (190 m³/d) of desalted groundwater. The concentrate flow of 4.6 gpm (17.4 l/m) was used in the self-sealing research program. HMUD was under a moratorium for providing additional water service hookups because the utility's wells produce groundwater with a

1

TDS concentration of approximately 1250 mg/l, which exceeds Texas drinking water standards. The desalted groundwater from this project provided a low TDS source of water which, during the winter months, brought the mixed (desalted and raw water) system supply to an overall TDS of less than 1000 mg/l on several occasions.

During the first year of this two-year project a dual membrane desalination system with intermediate softening between the first and second stages was fabricated and installed at Homestead in a 30- by 30-foot steel building. Additionally, two 12,000 gal (45.4 m^3) and two 4,000 gal (15.5 m^3) storage tanks were plumbed into the system for handling permeate and reject flows. Three 40 x 180 x 2.5 foot deep (12.2 x 55 x 0.76 m) storage ponds were also constructed for the segregation and storage of concentrate and ion exchange waste regenerate, and for evaporation testing. Excess concentrate was diverted to an on-site infiltration basin.

The system began operation in May of 1997 and was operated until June of 1998. During this time 8,000,000 gal (30,300 m³) of desalted water were produced for use by HMUD.

Research on the use of brine for self-sealing of concentrate evaporation ponds has produced very encouraging results in laboratory permeameters and in the field. Hydraulic conductivities of 10^{-7} centimeters per second were achieved in the laboratory. More importantly, the relationship between application methods, number of applications, type of chemical precipitate and initial hydraulic conductivity were statistically analyzed, The utilization of synthetic brines that form a calcium carbonate (CaCO₃) precipitate performed better than calcium sulfate (CaSO₄) precipitate at reducing soil hydraulic conductivity. The application of laboratory findings to field permeameters at HMUD produced positive results but much work remains.

The potential for greatly reducing the cost of concentrate disposal and utilization through the use of self-sealing evaporation ponds is high. Future research should focus on low cost methods for reducing the initial permeability of the soil, precipitate application and curing methods, liner stability and soil pretreatment methods.

2

2.0 Background and Research Methodology

This chapter reviews the history of the Homestead Municipal Utility District (HMUD), the impact of the project on the District, the origin and need for self-sealing evaporation basins, and an overview of laboratory and field experimental methodology used during the course of the research. Photographs of the site and membrane system are shown in Figures 2-1 through 2-4.

2.1 Homestead Municipal Utility District (HMUD)

HMUD is located on the far east side of El Paso in El Paso County on U.S. Highway 62/180 near the Hueco Mountains. The area has a current population of about 8500 of which approximately

5000 people reside



Figure 2-1. View of HMUD showing evaporation ponds in the foreground and the membrane system building on the left.

in the Homestead community. The community is comprised primarily of residential development with much vacant land. HMUD has been under a moratorium for the provision of new connections to its water distribution system because the well water does not meet the Texas maximum total dissolved solids (TDS) limit of 1000 mg/l.

Homestead Municipal Utility District No. 1 was created by Senate bill No. 1465 on May 25, 1985. In 1993, under the guidance of a new Board of Directors and General Manager Ronald B. Rodenhaver, the district initiated a program to come into compliance



Figure 2-2. Building at HMUD housing the membrane treatment system.

with state laws. (Rodenhaver, 1998)

Through the U. S. Department of Agriculture's (USDA) Rural Economic and Community Development Department and Texas Water Development Board, funding was obtained to construct a 250,000 gal (946 m³) elevated storage tank, a 248,000 gal (937 m³) ground storage tank, new pumping



Figure 2-3. Membrane treatment system showing first stage nanofiltration in the background and second stage in the foreground.

facilities, and a completely new distribution system with fire hydrants. Most of these improvements were completed during 1996. The Texas Water Development Board and USDA's Rural Development Department provided the funding for a 24-inch (61-cm)



Figure 2-4. Student researcher standing next to second stage pressure vessels.

transmission main from the El Paso Water Utility (EPWU) to HMUD. This line became operational in late 1998. The construction of this line allows the distribution of water with a TDS less than 1000 mg/l to HMUD residents.

The membrane system constructed at HMUD headquarters, as part of this pilot investigation, provided a low TDS supplementary water from May 1997 to July 1998. This water lowered the TDS of blended water and, at times during low demand periods in the winter, brought the TDS levels to less than 1000 mg/l in the distribution system. Figure 2-5 shows the blend of HMUD well water and project permeate needed to produce a blended water with a TDS of 1000 mg/l or less for distribution.

The pilot system has the capability of producing up to 100,000 gpd (380 m³/d). However, only half of the membrane elements were placed in the pressure vessels, limiting the production capacity to 50,000 gpd (190 m³/d). Therefore, two curves are shown in Figure 2-5: one for the production of 50,000 gpd (190 m³/d) and the other for 100,000 gpd (380 m³/d). When HMUD water usage was less than approximately 110,000 gpd (416 m³/d) at a TDS of 1400 mg/l and the pilot system was producing 50,000 gpd (190 m³/d) at a TDS of 100 mg/l, the final distribution water TDS dropped below the 1000 mg/l Texas limit.



Figure 2-5. HMUD Blended TDS at two permeate production rates

The process of finding and selecting HMUD as a project site is given in a previous report entitled "Preliminary Research Study of a Water Desalination System for the East Montana Area Subdivisions of El Paso County, El Paso Texas" (Turner, *et* al., 1995).

2.2 Sealing of Evaporation Basins

A major cost element of evaporation ponds is the geomembrane liner. An alternative concept is to attempt to use the chemicals in desalting concentrate to seal the bottom of the evaporation ponds -referred to as self-sealing. The idea originated from observations of playa lakes that sometimes have low permeability bottoms resulting from precipitation of evaporated salts. Figure 2-6 is a picture of Lake Lucero, the playa lake serving as a source for the gypsum dunes at White Sands National Monument. Precipitated salts appear to have sealed the bottom of this and other similar playa lakes.

Research in self-sealing technology, utilizing concentrate as a resource, was needed. A salt liner could theoretically be formed in a number of ways. The desired result is a precipitate liner that reduces the soil permeability to a magnitude of 10^{-7} cm/s or less. The advantage of employing self-sealing salt liners are (1) the cost, the concentrate contains the agents to induce mineral salt precipitates, and (2) the liner is self-repairing.



Figure 2-6. Lake Lucero is a playa lakebed located in southern New Mexico.

In order to conduct research on self-sealing the reject water produced by the Pilot Desalination Plant (PDP) system and the ion exchange units were segregated and stored in adjacent evaporation ponds. The ion exchange waters contain most of the calcium and magnesium used to form carbonate and sulfate mineral salts. The purpose of segregating the brines was for (1) field experiments on self-sealing, (2) studies on brine suitability for use as salinity-gradient solar pond (SGSP) medium, and (3) work on enhanced evaporation.

2.2.1 Laboratory Experiments

Laboratory testing began in the fall 1997. Permeameters as shown in Figure 2-7 were constructed to perform constant-head, steady state permeability tests (according to ASTM guidelines). Silica sand (base soil) for each soil type) or a silica sand-bentonite clay mixture were used for sealing tests. The experiments had four main variables: (1) soil permeability, (2) type of chemical solution, (3) addition



Figure 2-7. Laboratory permeameter.

technique, and (4) number of treatments or doses. Soil permeability was tested initially to

obtain baseline permeability: silica sand, 10^{-2} cm/s, and silica sand and bentonite clay mixture, $10^{-3.5}$ cm/s.

Chemical solutions of 4.33 M calcium chloride (CaCl₂), 3.43 M diammonium sulfate ((NH₄)₂SO₄), and 0.59 M sodium carbonate (Na₂CO₃) were prepared for addition to the soil column as shown in Figures 2-8 and 2-9. The addition technique refers to the method by which single-salt solutions were applied to the soil column, either as a combined or binary solution (CaCl₂- Na₂CO₃ or CaCl₂-(NH₄)₂SO₄) or in an alternating pattern (CaCl₂ and Na₂CO₃). The



Figure 2-8. Inside of laboratory permeameter.

number of treatments or doses describes how many times the chemical solutions were applied to the soil column. Experiments typically ran for one to three weeks with chemical additions taking two to three days to form the salt layer above the soil column.

2.2.2 Field Experiments

The second stage membrane concentrate and the ion exchange resin regenerate were stored separately in two of the three lined evaporation ponds (see Figure 2-10). The water softener added between the first and second stages lowered calcium and magnesium concentrations (hardness ions) entering the second stage. The second stage concentrate contained predominantly sodium, chloride, and sulfate ions; whereas the ion exchange regenerate contained sodium, calcium, magnesium,



Figure 2-9. Extruded treated sand from two laboratory permeameters.

and chloride

The concentration of water in the evaporation ponds varied with seasonal evaporation rates and operation of the membrane treatment system. The concentrate and regenerate brines from these ponds were used as feedstock for the



Figure Z-10. Lined evaporation basins.

self-sealing experiments conducted in field permeameters which were constructed adjacent to the evaporation basins (see Figure 2-1 1).

Four field permeameters were constructed by recycling two steel tanks from a previous research project. The permeameters were underlaid by a coarse sorted gravel to facilitate drainage. The water eventually drained out of the gravel into the sump where the experimental



Figure 2-11. Field Permeameters.

saline vegetative wetland was located.

Field treatments were prepared by mixing ion exchange regenerate with sodium carbonate forming a milky white precipitate of calcium carbonate. The solution was mixed in a plastic trash can then pumped and sprayed on the soil surface. A variety of treatments



Figure Z-12. Application of sealing solution and view of the bottom of an infiltration testing ring after drying.

were tried including a) seeding the soil with sodium carbonate followed by watering, b) spraying the precipitate on the top of the soil surface, c) alternating layers of treatment with addition of more sand, and d) mixing bentonite with the precipitate and spraying the resultant mixture. Photographs of the spray application and a dried layer of precipitate are shown in Figure 2-12.

3.0 Conclusions and Recommendations

Significant progress was made towards the development of a self-sealing technology for concentrate evaporation and storage ponds. Concentrate self-sealing technology will eventually become the method of choice because it is cost effective.

The humanitarian outcome of this work was the production of over eight million gallons of high quality drinking water that helped improve the lives of the five thousand residents of the Homestead Municipal Utility District.

3.1 Conclusions for HMUD Pilot System Design, Construction and Operation

System design of the two-stage membrane system with intermediate softening was accomplished with the assistance of Fluid Process Systems, Inc. (FPS). The design of the electrical system was done with the assistance of RBM Engineering of El Paso and Consolidated Electrical Distributors, Inc. Site **layout**, building design and layout, electrical system, data recording, piping systems and site experimental setup were coordinated through the use of Microsoft Project.

Construction required one year. The students researchers did not have prior construction experience and the faculty had minimal experience. An experienced technician from UTEP's salinity gradient solar pond test facility in North El Paso provided valuable assistance throughout the construction phase of the project. Communication and coordination were the biggest challenges during construction.

Operation of the system was never routine due to the research aspects of the project and the multiple objectives. Additionally, wells other than the two normally used by HMUD were available for use when problems were encountered with wells #11 and #12. The alternate wells produced water with much higher TDS. During periods when the higher TDS wells were placed on-line, changes in operation were dramatic because of the need to adjust system feed pressure and modify ion exchange operation. HMUD personnel were very helpful whenever possible. Their ability to assist was somewhat limited by the complexities of the system and the intensive nature of the data collection. **Training** was provided by Fluid Process Systems, Inc. The training took place over the course of one day. One week of training would have been optimal. The system was designed to operate 24 hours a day, 7 seven days a week throughout the year. Scheduling students for data collection and operation was difficult but was accomplished with weekly meetings, and with the assistance of Jesus Moncada a doctoral candidate assigned as the chief coordinator.

Materials handling can be problematic. Moving large quantities of salt and acid requires two people on-site, and safety had to be continually stressed. There were no significant injuries during the course of the project.

Coordination with HMUD required meetings with the district manager and the Board of Directors. The HMUD Board of Directors was supportive and helpful throughout the life of the project.

Publicity from the project was very positive. TV stations and the local newspaper ran several articles on the project which gave positive accounts of UTEP, EPWU and the USBR efforts to provide water for the colonias.

3.2 Conclusions for Self-sealing Laboratory Work

A factorial experimental design was used to reduce the number of experiments needed to obtain statistically reliable data. Four test factors: 1) soils of different initial hydraulic conductivity, 2) different chemical solutions, 3) different chemical application techniques and 4) number of applications, were investigated. The objective was to reduce the hydraulic conductivity of the test soil. The experiments were statistically analyzed using *JMP* computer software (developed by the *SAS* Corp.). The following conclusions can be defended based on the results of these experiments:

1. The utilization of chemical solutions that react to form a $CaCO_3$ precipitate performed better than $CaSO_4$ precipitate at reducing soil hydraulic conductivity. Irrespective of which test factor was altered, all experiments with $CaCO_3$ deposition had lower final hydraulic conductivity. This fact may have been affected adversely by possible dissolution of $CaSO_4$ precipitate.

- 2. Initial hydraulic conductivity was deemed significant in reducing hydraulic conductivity. Soils with an initial low hydraulic conductivity produced a soil with a lower final hydraulic conductivity after chemical application.
- 3. The number of applications test factor could be significant. The more times a chemical solution was applied, the more precipitates would be deposited. However, in these experiments, number of applications may have not been differentiated enough to notice this test factor and its possible importance.
- 4. Several prominent problems existed in testing. The first problem was possible $CaSO_4$ dissolution. The quantity of $CaSO_4$ precipitate appeared to shrink when comparing pre- and post-hydraulic conductivity tests measurements. In addition, some experiments using $CaSO_4$ had a slight increase in hydraulic conductivity. This might again indicate that $CaSO_4$ was going into solution during the hydraulic conductivity testing. Another problem in testing was not compacting the experimental soil (Mannion, et al., 1968). The experimental soil may have been affected by undesirable volume changes like swelling during chemical application. Also, if the soil was compacted more rigorously, the average pore size would have been reduced. This pore size reduction might have enabled deposited chemicals to bridge and till voids more easily.

3.3 Recommendations for Self-sealing Laboratory Work

Based on these experiments, the following recommendations are made for self-sealing evaporation pond research:

- I. Further research in the formation process of $CaCO_3$ and $CaSO_4$ is required. These precipitates may be affected more acutely by time between chemical applications, pH of chemical solutions, temperature and concentration of chemical solutions.
- 2. Sodium silicate, lignin pitch and/or methyl cellulose should be tested. Sodium silicate can be set by calcium ions in brine and the hydraulic conductivity can be reduced further by subsequent deposition of precipitates. Lignin pitch, with the addition of alum, is also capable of reducing the hydraulic conductivity. Finally, methyl cellulose applied to a soil will only need water to gel and also effectively reduces hydraulic conductivity. These materials were demonstrated to be feasible in the mid 1960's (Mannion ef al., 1968).
- 3. Hydraulic conductivity of soil is affected by its compaction. Soils compacted to optimum density may assist in chemical deposition in voids.

3.4 Conclusions for Self-sealing Field Work

All field experiments reduced field permeability significantly, but not to the extent desired. Once again, initial permeability was shown to be a significant factor in self-sealing testing. Field permeameters were four feet in diameter and required large quantities of brine for the testing. The ion exchange resin regenerate contained the multivalent cations from the well water and was very effective in producing the binary system precipitates for the self-sealing research. The waste regenerate was mixed with sodium carbonate to form calcium carbonate precipitate.

The application of laboratory findings to field permeameters at HMUD produced permeabilities near 10^{-4} cm/s. The goal for permeability reduction was 10^{-7} cm/s. Based on both field and laboratory experiences, additional work should be conducted in the laboratory and once again tested in the field. Transfer of laboratory protocol to the field was effective.

3.5 Recommendations for Future Self-sealing Work

- 1. The effects of initial permeability on self-sealing should be further investigated. Initial permeability was shown to have an effect on self-sealing but the extent of this effect in terms of reducing time to achieve the desired permeability is not known.
- 2. Methods for reducing initial permeability should be investigated. The traditional method of using soils with high clay content can be very expensive when clay is not readily available. Initial permeability can be reduced through the application of materials like sodium silicate. Slowing the movement of the supersaturated brine through the uppermost layer of the pond appears to enhance the precipitation of the salts, thereby reducing permeability.
- 3. Application techniques need to be investigated. Cycles of application followed by drying periods or alternate application of solutions may play a role in the rapid reduction of permeability.
- 4. Stability of self-sealing liners that are developed should be evaluated.
- 5. The potential for using historical dry lake beds should be investigated. Many of these may already have very low initial permeability.
- 6. The long-term feasibility of using precipitate salts as by-products should be evaluated.

4.0 HMUD Facility

4.1 Design of the HMUD Pilot Desalination Plant

The research objective of the HMUD pilot desalination plant was to produce a low volume, high TDS concentrate suitable for self-sealing research. The operational objective was to provide HMUD with a high quality permeate. The volume of the permeate had to be large enough to significantly reduce the TDS concentration of HMUD saline well water while the concentrate volume had to be small enough to dispose of on-site. These objectives had to be accomplished while staying within a tight budget.

Recovery had to be high because all concentrate had to be disposed of on-site through evaporation and infiltration. Deep well injection was too costly. The available land area was limited to approximately one acre so the proposed on-site evaporation system had to be used primarily for concentrate research and not disposal HMUD had a holding basin for use when either of their storage tanks needed to be emptied for maintenance. The basin was judged suitable for infiltration of a continuous flow of 4 to 5 gpm (0.25 to 0.32 l/s) of concentrate. The soil is a highly permeable sandy loam.

The concentrate flow rate served as the limiting factor in system design. The largest capacity pilot system that could be supported by the level of funding available was determined to be approximately 37.5 gpm (2.37 l/s) based on a 50 gpm (3.15 l/s) feed and a 75% recovery. This flow rate would produce 12.5 gpm (0.79 l/s) of concentrate which would have been too high for on-site disposal. A second stage (stage II) was added to the system to process the first stage concentrate. This reduced the volume of the total system concentrate while increasing TDS concentration of the stage II concentrate. Since concentrate from stage I was used as feed for stage II, removal of calcium was necessary to prevent scaling of stage II membranes. A salt-regenerated cation exchange system was selected for softening of the stage I concentrate prior to being used as feed for the stage II system. This served two purposes: first, scaling potential for the second stage was greatly reduced; and second, the multivalent cations were removed for use in self-sealing experiments.

An iterative procedure was used to arrive at the correct combination of flow rates to

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meet system goals. Figure 4-1 is a schematic of the system showing the process units and flow rates. The two-stage membrane system has an input flow rate of 3 1.2 gpm (1.97 l/s), permeate production rate of 26.6 gpm (1.68 l/s) or 38,300 gpd (145 m³/d), and a stage II concentrate (reject) flow of 4.6 gpm (0.29 l/s) or 6,770 gpd (25.6 m³/d). The total membrane system recovery is 85%. The first stage recovery is 75%. Figure 4-1 shows a system inflow of 63.7 gpm (4.02 l/s) with a by-pass flow of 32.5 gpm (2.05 l/s) for mixing with the membrane system permeate to produce a total blended flow rate of 59.1 gpm (3.73 l/s) or 85,100 gpd (322 m³/d).

The design TDS concentration of the permeate and final blended product water was 40 mg/l and 670 mg/l, respectively. The typical well water chemical composition is shown in Table 4.1, Using the membrane manufacturer's software, the predicted concentrations for sulfate, chloride, bicarbonate, calcium, and magnesium in the final blended product water were 109, 286, 48, 29, and 4 mg/l, respectively.

4.1.1 Membrane Selection

Hydranautics introduced a new low-pressure membrane, the ESPA series, near the beginning of this project. Specifications for the ESPA membrane are shown in Table 4-2. The new membrane is different from the low-pressure membranes originally planned for use with the PDP. The **8040-UHY-ESPA** has the high salt rejection characteristics of the CPA2 series, but operates at a significantly lower operating pressure of 150 psig (1.05 MPa). The **8040-CPA2** and **4040-CPA2** membranes both operate at a feed pressure of 225 psig (1.55 MPa) (Hydranautics 1994), 75 psi higher than the 8040 ESPA membrane. Specifications for the CPA2 series membrane are shown in Table 4-3.

The minimum salt rejection for the ESPA membrane is 99.0%; whereas, the CPA2 is 99.5% (Hydranautics, Inc. 1994). As a result of the new membrane introduction, the PDP design uses five 8040-ESPA elements for stage I and five 4040-CPA2 membranes for stage II. The design modifications were performed to maintain the flow scheme of the original design, as diagramed in Figure 4.1. Tables 4-4 and 4-5 list the chemical quality of the stages I and II permeate and concentrate streams, respectively.



Figure 4-1. Schematic of pilot desalination system.

Constituent Name	Well No. 11 ¹	Well No. 12 ²	Blended ⁵
bicarbonate (mg/l ion)	76	90	83
carbonate (mg/l ion)	0	0	0
calcium (mg/l ion)	61	44	53
chioride (mg/l ion)	585	433	509
fluoride (mg/l ion)	1	1	1
magnesium (mg/l ion)	9	7	8
nitrate (as N) (mg/l)	0	0	0
potassium (mg/l ion)	0	0	0
sodium (mg/l ion)	408	336	372
sulfate (mg/l ion)	215	180	198
total hardness (as CaCO ₃)	191	137	164
pH	7	7	N/C
diluted conductivity (µmhos/cm)	2704	2096	2400
total alkalinity (mg/l CaCO ₃)	62	74	N/C
dissolved solids	1324	1050	1238
phenol alkalinity (mg/l CaCO ₃)	0	0	0
arsenic (mg/l ion)	<0.010⁴	0.018	N/C
barium (mg/lion)	0.053	0.052	N/C
cadmium (mg/lion)	<0.0050⁴	<0.00504	N/C
chromium (mg/l ion)	< 0.0204	<0.0204	NC
copper (mg/l ion)	<0.020⁴	0.05	N/C
iron (mg/l ion)	0.14	0.08	0.11
lead (mg/l ion)	<0.0204	<0.0204	N/C
manganese (mg/l ion)	<0.0204	<0.0204	N/C
mercury (mg/lion)	<0.00020⁴	<0.000204	N/C
selenium (mg/l ion)	< 0.0104	<0.010⁴	N/C
silver (mg/l ion)	< 0.0104	<0.010⁴	N/C
zinc (mg/lion)	0.08	0.12	N/C
silica (mg/l as SiO ₂) ³	15	15	15

 Table 4-1: Source Well Water Quality for Wells No. 11 & 12.

N/C Not calculated as blended water quality.

- 1. Well no. 11 water was sampled 8/5/92 with results reported 9/16/92.
- 2. Well no. 12 water was sampled 8/14/92 with results reported 9/23/92.

3. Value for silica based on 1996 analyses.

4. Value is below the detection limit for that ion.

5. Blended water quality was determined by a Fluid Process Systems, Inc. spreadsheet software **that** calculated the blended water chemical quality.

Membrane Characteristic	Value, metric (US customary)
Permeate flow (nominal)	45.4 m ³ /day (12,000 gpd)
Salt rejection (average)	99%
Configuration	spiral-wound
Membrane polymer	composite polyamide
Nominal area	37 m ² (400 ft ²)
Normal operating pressure	1.05MPa (150 psig)
Maximum applied pressure	4.16 MPa (600 psig)
Maximum feed flow to element	17 m ³ /hr (75 gpm)
Maximum feed water turbidity	1.0 NTU
Average specific flux rates (well water SDI < 2)	14 - 18 gfd

Table 4-2: 8040-ESPA Membrane Characteristics.

Table 4-3: 4040-CPA2 Membrane Characteristics.

Membrane Characteristic	Value, metric (US customary)
Permeate flow (nominal)	8.5 m³/day (2,250 gpd)
Salt rejection (average)	99.5%
Configuration	spiral-wound
Membrane polymer	composite polyamide
Nominal area	7.9 m² (85 ft²)
Normal operating pressure	1.55 MPa (225 psig)
Maximum applied pressure	4.16 MPa (600 psig)
Maximum feed flow to element	17 m³/hr (75 gpm)
Maximum feed water turbidity	not given
Average specific flux rates (well water SDI < 2)	not given

Constituent Name	Permeate (mg/l ion)	Concentrate (mg/l ion)
calcium	0.3	209.1
magnesium	0.0	31.9
sodium	10.5	1457
carbonate	0.0	0.1
bicarbonate	3.9	320
sulfate	1.3	786
chloride	13.5	1996
fluoride	<0.5	2.7
nitrate (as N)	0.1	1.4
silica	0.4	119
TDS	30.1	4923

Table 4-4: Stage I Permeate & ConcentrateChemical Quality ESPA Projection.

Table 4-6 lists the chemical quality of the final blend water produced from the modified design. As stated earlier, the two flows are to be blended with a diverted feed raw water stream to increase water production and remain within the 400-600 mg/l of TDS range. As shown in Table 4-6, the chemical quality of both the permeate and final blended product water are within the State of Texas TDS limit.

One design criterion was to reduce the amount of concentrate produced. Concentrate disposal is problematic for inland, semi-arid desert regions because the concentrate cannot be discharged directly without adversely affecting the sensitive desert ecosystem. Stage II used CPA2 series membranes that operate at higher pressures than the ESPA series. This reduced stage II concentrate to a flow rate of 4.6 gpm (0.29 l/s). Ion exchange softening reduced the amount of calcium present in the concentrate to prevent calcium scaling, predominantly $CaSO_4$. Stage II feed water was also pretreated with an anti-sealant to provide additional protection against calcium and silica scaling.

Concentrate production, based on a 24-hour operation day, was 6,768 gal (25.6 m^3). The concentrate volume was relatively substantial given the one acre experimental site size limit. This concentrate volume would have caused problems because an evaporation

Constituent Name	Permeate (mg/l ion)	Concentrate (mg/l ion)
calcium	0	35
magnesium	0	0
sodium	40	2940
carbonate	0	0
bicarbonate	124	524
sulfate	5	1310
chloride	49	3290
fluoride	0	4
nitrate (as N)	0	2
silica	1	197
TDS	110	8302

Table 4-5: Stage II Permeate and Concentrate Chemical Quality.

pond volume storage of 27,140 ft³ (769 m³) would have been needed to store one month's production of concentrate. Evaporating the concentrate produced without accumulating any volume, given the lowest monthly average evaporation rate of 1.96 in/month (5.0 cm/month), would have required an evaporation pond with the surface area of 165,520 ft² (15,377 m²). This is approximately 4 acres, considerably larger than the land available at HMUD.

The predicted concentrate quality (see Table 4-5) was 8,300 mg/l of TDS. Sodium, sulfate, and chloride concentrations were predicated at 2,940 mg/l, 1,310 mg/l, and 3,290 mg/l, respectively. A high sodium concentration in the product results from the exchange of sodium ions in the ion exchange softening process for hardness. Sodium, which has a higher solubility than hardness forming ions, is much less likely to foul the membranes.

4.2 Construction of the HMUD Pilot Desalination Plant

The construction of the PDP covered a period of approximately one (1) year. Fluid Process Systems, Inc. completed construction of the PDP in March 1997. The PDP was installed in April 1997 and operation of the facility began in **late** May 1997. Over the course of the year several milestones were achieved before the facility became operational. The milestones are described in the following paragraphs.

Constituent Name	Combined Permeate	Final Blended Product (mg/l ion)
A REAL PROPERTY OF A READ REAL PROPERTY OF A REAL P	(mg/l ion)	
calcium	0.2	29
magnesium	0.0	4
sodium	10	210
carbonate	<0.1	¹ <0.1
bicarbonate	3.6	47
sulfate	1.2	110
chloride	12	290
fluoride	<0.1	0.4
nitrate (as N)	0.02	0.2
silica	0.3	0.2
	-	· · · · · · · · · · · · · · · · · · ·
Flow rate (gpm)	27	59

Table 4-6: Final Product Water Chemical Quality of the PDP.

- The three-bay concentrate storage pond was constructed during the summer of 1996. Several designs were evaluated. The final design used wooden walls to support the liner instead of a conventional soil embankment. The pond constructed was 120' x 180' x 2.5' deep subdivided into three bays each 40' by 180'. Three high-density polyethylene (HDPE) liners, each sized for the individual bays, were installed in August 1996.
- The PDP housing or facility building was contracted out in September 1996 and construction began in September/October 1996. The steel building was erected on a 2-foot thick concrete slab to support the weight of the building, the PDP and two 12,000 gal (45.4 m^3) fiberglass water tanks.
- . Installation of the PDP, including electrical wiring, plumbing and structures, began in March 1997 and continued until mid-May 1997. Construction of a suitable power supply began early in 1997 and was a pivotal milestone to PDP star-up.
- Construction of the self-sealing basin field and saline vegetative wetland began in late summer of 1997.
- The entire test facility was constructed with student labor supplemented by staff and faculty assistance.
- The PDP met all design criteria at the time of installation and start-up

5.0 Results and Discussion

The following section describes the performance of the PDP over the research period starting in May 1997 ending in June 1998. The operational performance and cost of the PDP at Homestead are described in detail in the next two sections. Section 5.1 reviews the operating performance of the PDP. Section 5.2 reviews the costs of the construction, installation and operation of the PDP and concentrate management facilities. Costs related to research, at the site or in the laboratory, were not included in the economic evaluation.

5.1 Desalination Plant System Operation

As stated earlier, the PDP began operation in late May 1997. The operating parameters recorded over approximately fourteen months are listed in Table 5-1. Highlighted parameters were used for normalization of the results.

Operating Result	
Normalized permeate flow (gpm)	
Average membrane normalized flux (gpd)	
Average membrane normalized salt rejection (%)	
System normalized salt rejection (%)	
Calculated Operating Parameters (based on above results)	
System normalized salt passage (%)	
System recovery (%)	

Table 5-1: Operating Parameters and Calculated Parameter Variables.

The three primary operating variables of concern were system temperature (T), feed pressure (P_f) and feed conductivity (C_f).

Graphs showing daily operating parameters have significant gaps in the data. These gaps are a result of system downtime caused by system failure, piping or plumbing repair, or system re-engineering. The gaps are usually of one to four week time periods depending on the cause.

The feed temperature trends for both stages I and II are caused by seasonal

temperature variation as shown in Figure 5-1

Figure 5-2 shows the daily feed pressures for stage I and stage II, respectively. At start-up, stage I feed pressure remained between 90 to 100 psig. During October 1997, an abrupt spike in feed water conductivity was recorded when a highly saline well was brought online. Typical feed water conductivity remained constant within a range between 1,700 to 2,500 μ S/cm. The spike registered a magnitude twice that of the typical feed conductivity, approximately 4,000 μ S/cm.

The spike caused a sharp rise in feed pressure for stages I and II, and the feed conductivity for stage II (stage I reject). An increase in feed pressure was necessary to 'compensate for the high TDS feed water. There was a lag in adjusting the feed pressure so conductivity and feed pressure do not parallel each other during this period.

The large pressure fluctuations shown in Figure 5-2 between March and June 1998 were attempts to maintain permeate flow. Scaling of the elements made operating stage II difficult during this time period. Cleaning of stage II took place in mid-July 1998. Afterwards, feed pressure remained steady between 130 and 150 psig. Flow rate for stage II returned to its original operating level.

Feed conductivity for both stage I and II remained fairly consistent (see Figure 5-3), except for the conductivity spike that occurred near the end of October 1997.

Permeate flows for stage I and stage II were designed for 23.4 gpm and 3.1 gpm, respectively. Figure 5-4 shows both the normalized and non-normalized permeate for both stages. In both figures, normalized and non-normalized permeate flow rate (gpm) roughly parallel each other. After star-up, the membrane elements experienced a gradual flux decline. The elements eventually stabilized slightly below their design flow rates. Feed pressure was not increased to raise the permeate flow rate back to design specifications.

Feed pressure was adjusted to compensate for the increase in feed water TDS as shown in Figure 5-2. Pressure was increased later after the conductivity spike was observed. Stage II (Figure 5-2) also shows an increase in flow rate as feed pressure was raised to compensate for the higher TDS concentration in November. After November

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Figure 5-1. Daily feed temperature for stages I and II.





Figure 5-2. Daily feed pressure for stages I and II




Figure 5-3. Daily feed conductivity for stages I and II.





Figure 5-4. Average monthly permeate flow.

1997 the flow rate gradually declined as a result of scaling of the elements. Feed pressure was increased to aid in permeate production. Scaling became a larger factor leading to permeate flow decrease. The reason for stage II scaling was a problem with the ion exchange units. The two ion exchange units were set to regenerate after a programmed time elapsed. Several adjustments were made; however, the resin was not regenerated frequently enough and excess hardness (calcium and magnesium) was allowed to pass.

An objective of this project was to provide high quality permeate to the residents of Homestead. Figure 5-5 shows the permeate production of stage I and II, respectively. Small production months were a result of system downtime.

Salt rejection performance of the membranes is shown in Figure 5-6. Salt rejection and salt passage for the ESPA and CPA2 membranes are presented graphically in the figure. Salt rejection and salt passage remained steady until November 1997. After November the membranes in both stages began to experience a decline in salt rejection.

Stage I shows a sharp decrease in salt rejection as a result of chlorine exposure in early April which caused considerable damage to the stage I membrane elements. The elements eventually stabilized at much lower salt rejection levels.

Figure 5-7 shows the percent recovery trends for stages I and II and the system as a whole. System percent recovery falls between 70 to 80% over the year with a gradual decrease because of scaling. The peak in the trend is indicative of the feed pressure adjustments to compensate for the conductivity spike observed during the time period.

5.2 Pilot Desalination Plant System Costs

The costs are divided into three categories: (1) research costs, (2) capital costs, and (3) operation and maintenance costs. Costs incurred for research purposes (e.g. permeameters, standards, non-pretreatment chemicals, and tools) were not included in the economic analysis.

Capital and brine disposal costs are represented as a single cost. Capital costs (Table 5-2) include all materials, equipment and other costs that were incurred only once during the project duration. This includes the following: the desalination units (stage I and stage





Figure 5-5. Average monthly permeate production.





Figure 5-6. Average monthly salt rejection.



Figure 5-7. Average monthly percent recovery

II), building and related facilities, membrane elements, and storage tanks. Concentrate disposal (Table 5-3) includes capital, non-capital and operational & maintenance (O&M) costs. The reason for treating this separately is to compare this facility with other available and conventional concentrate disposal technologies.

Item Description	Unil Cost	No. of Units	Extension
Facility (building, concrete slab, driveway, fence)	\$28,500	1	\$28,500
Land (1 year lease)	\$36,000	1	\$4,000
Electrical (transformer, power cables, power switch)	\$7,500	1	\$7,500
Stage I & II (RO system and pretreatment equipment)	\$83,691	1	\$83,691
Stage I & II Installation	\$13,175	1	\$13,175
Stage I & II ESPA and CPA2 membrane elements	\$6,359	1 (all inclusive)	\$6,359
		Total Capital Costs:	\$143,225

Table 5-2: Itemized Capital Costs for Pilot Facility.

Item Description	Unit Cost	No. of Units	Extension
Three high-density polyethylene liners	\$5,400	1 (all inclusive)	\$5,400
Pond materials (wood, nails, concrete)	\$2,000	1	\$2,000
Equipment (pawer tools, shovels)	\$1,200	1	\$1,200
Equipment rental (auger, front loader, compactor)	\$30	45 working days	\$1,350
Labor (construction: estimated as 4 persons working all 45 days for 6 hours per day @ \$12.00 per hour)	\$15	1080 man hours	\$16,200
Monitoring well	\$1,000	1	\$1,000
	: To	atal Brine Disposal Costs:	\$27,150

Table 5-3: Itemized Costs for Brine Disposal.

Operation and maintenance (O&M) costs (Table 5-4) include non-capital, chemicals, consumable materials, electricity, and labor. O&M costs are represented as a unit cost per 1,000 gal of permeate produced. Because the facility was intended for research, capital and brine disposal costs are not amortized over time. Non-capital costs include costs for computers, printers, laboratory and field analysis equipment, tools and power equipment, and consumable materials over \$1,000. Office materials, laboratory chemicals, and other materials needed for everyday administration and operation of the project are categorized under consumable materials. Electrical costs are not indicative of a low-pressure desalination system. Both stages were equipped with high pressure pumps that produce an average pressure of 310 psig. The pressure was throttled down to a design range (specific to each stage) to mimic a typical low-pressure system. In production applications the energy lost in throttling would be eliminated through use of variable-speed pumps.

Labor costs are calculated based on one person maintaining the facility for four (4) hours a day, five (5) days a week. A well-designed, well-built system would need very little maintenance and require daily system checks to ensure the system is meeting design specifications for permeate production.

Item Description	Unit Cost	No. of Units	Extension
Average monthly electrical costs	\$945	12 months	\$11,340
Muratic acid (hydrochloric acid, 20 Be: approximately 2 55-gallons drums used per month)	\$86	24 drums	\$2,060
Rock salt (regeneration of softener: pallets of 50 bags (50-lbs each) per month),	\$256	12 pallets	\$3,070
Anti-scalant (5 gallons pails, approximately 1 pail for 1.5 months)	\$249	6 pails	\$1,990
Maintenance (materials for repairs, re- engineering or other)	\$250	12	\$3,000
Labor (one person for 4 hours per day needed for 5 days of the week)	\$15	896 man hours	\$13,440
	\$34,900		
Total	7892000		
Unit	\$4.42		

 Table 5-4: Itemized Costs for O&M.

5.3 Self-sealing Results from the Laboratory

53.1 Evaporation Disposal Method

Inland desalination is limited by concentrate disposal costs (Gogineni, 1995; Koppula, 1995, Turner et al., 1995). In coastal regions, disposal of concentrate can be accomplished by discharge into the ocean. However, in arid regions of the interior U.S. and Mexico, concentrate cannot be discharged into a river or lake. This problem can be addressed by utilizing the concentrate as a self-sealing agent for evaporation ponds thereby eliminating the cost of a synthetic liner. If synthetic liners were eliminated, the cost of evaporation ponds could be decreased by as much as 30% (Jubran et al., 1996). Cost is not the only factor as liners have other flaws. Even with extreme care during installation and maintenance, leaks have been detected at most facilities that utilize liners (Laine and Miias, 1990). Despite most flaws being due to improper seaming and degradation, flaws will also occur with exposure to ultra-violet light, high energy radiation, oxidation, hydrolysis, microbial intrusion and chemical reactions (Rahman, 1995). Self-sealed evaporation ponds

because they have the potential to be self-healing and are formed from waste materials,

5.3.1.1 Theory

Soil consists of a collection of solid particles with voids in between them (Holtz et al., 1981). The solid particles can be made up of small or large grains of different minerals and organic materials. How a fluid flows through the soil, or more precisely the



Brine Concentrate



soil voids, is described as the hydraulic conductivity (k). Reducing the hydraulic conductivity of a soil involves reducing the void space. The void space can be changed either by mechanical (compaction) or chemical (cement, asphalt, lime, etc.) means and this ultimately leads to a densification of the soil mass. In laboratory and field experiments at Homestead, chemicals were applied directly to the soil mass to reduce void volume and thus reduce the hydraulic conductivity. The binary sealing method is illustrated schematically in Figure 5-8.

5.3.1.2 Sealing Method

The void space in a soil can be reduced through several means. Four variations are discussed herein, For example, the first method of sealing involves the settling of precipitated solids and the creation of a low hydraulic conductivity zone (Figure 5-9). In this case, deposition occurs in the mixing chemical solutions or in the combination of simple chemical solutions before entering the soil. Precipitates settle out by gravity and create a zone of lower hydraulic conductivity

The second method of sealing occurs when precipitated solids settle and then become a solid layer (Figure 5-9). This variation is similar to the previously discussed



Figure 5-9. Deposited precipitate layer.



Figure 5-10. Deposited solids clog soil voids at surface.

variation, where the chemical reaction occurs in the combination of chemical solutions before entering the soil. The difference between the two sealing variations is the creation of a distinguishable deposited solid layer and not just a zone of low hydraulic conductivity.

In the third sealing method, reduction in the hydraulic conductivity can be attributed to clogging of the soil voids (Figure 5-10). The chemical reaction and precipitation occur in the chemical solutions above the soil matrix. The solids infiltrate the upper most layers of the soil and the deposited solids fill the voids and obstruct the passage of water through the soil specimen.



Figure 5-11. Precipitates clogging voids within soil matrix.

The fourth and final method involves filling of the voids within the soil. Instead of precipitation occurring in the solution above the soil, precipitation originates in the voids and around the periphery of individual soil grains (Figure 5-1 1). As the minerals grow in size, the voids and water pathways are obstructed and the hydraulic conductivity decreases.

5.3.1.3 Factorial Experimental Design

When conducting research, experiments are performed to gather information from which conclusions are drawn. A traditional approach to experimentation would be to vary one parameter at a time. Depending on the complexity of topic, there may be many questions to be answered. A one parameter at a time approach requires excessive testing as the number of parameters increased. Experimental design methods were developed to reduce the number of experiments that would be required. There are many types of experimental designs that can be adapted for any situation. For the laboratory experiments, a factorial experiment design was chosen. Factorial experiments are the most commonly used multi-factor designs (Peterson, 1985).

The factorial experimental design for the laboratory consisted of combinations of four factors each with two levels. The combinations are such that each level of every factor occurs together with each level of every other factor. The number of experiments is the product of the number of levels of all factors. Again, there are four factors: initial hydraulic conductivity, type of chemical solution, chemical application technique, and number of applications. There are two levels of initial hydraulic conductivity, two levels of chemical solutions, two levels of chemical application techniques and two levels of the number of applications. The product of these levels was 2⁴ or 16 experiments. Although factorial experiment designs give estimates of the effects of factors, which are more precise than one at a time experimentation, sometimes it is necessary to replicate the design to obtain the desired precision

No.	Initial Hydraulic Conductivity	Type of Chemical Solution	Chemical Application Technique	Number of Applications
1	2.18 E-04	CaSO₄	Alternating	5
2	2.09 E-02	CaSO₄	Alternating	5
3	2.18 E-04	CaSO₄	Alternating	10
4	2.09 E-02	CaSO₄	Alternating	10
5	2.18 E-04	CaSO₄	Combined	5
6	2.09 E-02	CaSO₄	Combined	5
7	2.18 E-04	CaSO₄	Combined	10
8	2.09 E-02	CaSO₄	Combined	10
9	2.18 E-04	CaCO₃	Alternating	5
10	2.09 E-02	CaCO₃	Alternating	5
11	2.18 E-04	CaCO₃	Alternating	10
12	2.09 E-02	CaCO ₃	Alternating	10
13	2.18 E-04	CaCO₃	Combined	5
14	2.09 E-02	I CaCO₃	Combined	5
15	2.18 E-04	I CaCO₃	Combined	10
16	2.09 E-02	CaCO ₃	Combined	10

Table 5-5: Factorial Experiment Design.

The number of experiments needed to "do the job", N, is:

$$N = (7.5 \, \mathrm{\sigma}/\mathrm{\delta})^2 \tag{1}$$

where σ is the standard deviation of a single observation and δ is the size of an effect that we do not want to overlook (Erjavec, 1980). Because of the small units encountered in

hydraulic conductivity testing, a typical effect we do not want to overlook would have roughly the same magnitude as the standard deviation of a single observation and a value slightly greater than σ would be more realistic. If δ was chosen to be 200% greater than σ , then 14 experiments need to be done. This number was rounded to the nearest multiple of 16 experiments, which would be a total of 16 experiments. Therefore, no replication of experiments was required. In Table 5-5, a listing of the 16 experiments that were run is provided.

5.3.1.4 Chemical Relationships

In natural salt ponds such as alkaline lake beds, self-sealing layers can be formed along the pond bottom under specific conditions. The self-sealing layer is composed of different salts, such as sulfate, phosphate and carbonate compounds that seal the top of the soil medium or clog the voids further down in the soil medium to reduce the hydraulic conductivity. The factorial experiment design evaluated two chemical systems. The first system consisted of a combination of calcium chloride and sodium carbonate solutions (CaCl₂-Na₂CO₃) and deposited a carbonate mineral (CaCO₃). The second system was a combination of calcium chloride and diammonium sulfate solutions (CaCl₂-(NH)₂SO₄) and deposited an alkaline earth metal sulfate (CaSO₄).

In a carbonate system, carbon dioxide gas can react with water to form three carbonate system species: carbonic acid $(H_2CO_3^*)$, bicarbonate (HCO_3^-) and carbonate $(CO_3^{2^-})$. Note that $H_2CO_3^*$ represents the sum of the dissolved carbon dioxide gas and carbonic acid (Garrels, et al., 1965). In nature, these carbonate species exist in equilibrium and any change in concentration of one species will cause a shift in concentration in the other ions. When inorganic cations like magnesium (Mg^{2^+}) or calcium (Ca^{2^+}) are also present, precipitates such as magnesium carbonate or calcium carbonate may be deposited. In the following carbonate system equilibrium equations (2-5), carbon dioxide gas *can* be driven in a *certain* direction by factors including temperature and pH to react with an inorganic ion and furthermore precipitate out of solution.

$$\operatorname{CO}_2(\mathbf{g}) \Leftrightarrow \operatorname{H}_2\operatorname{CO}_3^*(\operatorname{aq})$$
 (2)

$$H_2CO_3^*(aq) \Leftrightarrow HCO_3^-(aq) + H^+(aq)$$
(3)

$$HCO_{3}^{-}(aq) \Leftrightarrow CO_{3}^{2-}(aq) + H^{+}(aq)$$
(4)

$$\operatorname{Ca}^{2+}(\operatorname{aq}) + \operatorname{CO}_{3}^{2-}(\operatorname{aq}) \Leftrightarrow \operatorname{CaCO}_{3}(\operatorname{s})$$
 (5)

The calcium carbonate precipitates out of solution and was expected to seal or clog soil voids. The precipitation of calcium carbonate requires a pH of 9.5 or higher and if this requirement is not met calcium bicarbonate ($Ca(HCO_3)_2$) may be produced (Reynolds, 1982).

In the CaCl₂-Na₂CO₃ experiments, calcium chloride (CaCl₂) and sodium carbonate (Na₂CO₃) were selected for the tests. First, calcium chloride was much more soluble in water than the previously used hydrated lime. This enabled calcium chloride solutions to have a higher concentration than the previous screening of calcium hydroxide solutions. Instead of a concentration of approximately 1 g/L of calcium hydroxide solution, the concentration of the calcium chloride solution was 50 g/L. Next, sodium carbonate was used for its specific carbonate species, CO_3^{2-} , instead of sodium bicarbonate, where the reaction was too dependent on excess lime for high pH requirements for calcium calcium carbonate precipitation. Therefore, the CaCl₂-Na₂CO₃ experiments produced calcium carbonate precipitate (Eqn. 6).

$$CaCl_{2}(aq) + Na_{2}CO_{3}(aq) \Leftrightarrow CaCO_{3}(s) + 2NaCI(aq)$$
(6)

Another anion present in most concentrates is sulfate (SO_4^{2-}) . Sulfate anions will react with inorganic cations, such as calcium (Ca^{2+}) , barium (Ba^{2+}) or strontium (Sr^{2+}) , in evaporation pond environments. During the screening process, hydrated lime $(Ca(OH)_2)$ was combined with hydrochloric acid (HCl) to make calcium chloride which was used as the inorganic cation donor. Next, a common fertilizer $((NH_4)_2SO_4)$ was utilized for its sulfate anion. In the final experiments, laboratory grade calcium chloride and diammonium sulfate were selected and the same reaction occurred (Eqn. 7).

$$CaCl_{2} (aq) + (NH_{4})_{2}SO_{4} (aq) \Leftrightarrow CaSO_{4} (s) + 2NH_{4}Cl (aq)$$
⁽⁷⁾

Calcium sulfate is naturally occurring in areas like White Sands, New Mexico. It is generally found in two forms, gypsum ($CaSO_4 \cdot 2H_2O$) and anhydrite ($CaSO_4$).

Modifications to calcium sulfate can occur during heating or variations in pressure but these modifications would not occur at the temperatures present in the laboratory, which indicates gypsum ($CaSO_4 \cdot 2H_2O$) will be present in the permeameters.

5.3.2 Laboratory Methods

5.3.2.5 Base Material Classification

In order to classify the base materials used for the latter experiments, the standard ASTM D 2487.92 was used (Table 5-6). This standard classifies soils according to the Unified Soil Classification System (USCS), which identifies coarse-grained soils, fine-grained soils, and highly organic soils. In addition, only parts of the standard were needed because the base materials contained negligible organics and fines (particles of specimen that will pass a 425 mm sieve and are retained on a 75 mm sieve).

Table 5-6: Simplified C	lassification Procedures.
-------------------------	---------------------------

No.	Soils Containing Less Than 5% Fines
1	Prepare soil specimens for particle size distribution and possible liquid limit and plasticity index tests
2	Prepare a set of sieves with the following sizes: 19.0 mm, 4.75 mm, 2.00 mm, 425 mm, 75 mm and any other sieve sizes needed
3	Report any material greater than 75 mm and determine the percentage by dry weight
4	With mechanical shaker, agitate sieves
5	Calculate percentage passing and/or retained on each sieve
6	Calculate coefficient of uniformity (C ₀) and curvature (C ₀)
7	Determine percentage of specimen that passed and was retained on 75 mm sieve
8	Follow USCS flow chart to determine group symbol and name

In Table 5-6 the terms, coefficient of uniformity and coefficient of curvature, are crude shape parameters used in classification systems. The coefficients are defined below in Equation 8 where D_{60} equals the grain diameter (mm) corresponding to 60% passing by weight, D_{30} equals the grain diameter (mm) corresponding to 30% passing by weight and D_{10} equals the grain diameter (mm) corresponding to 10% passing by weight.

$$C_{u} = \frac{D_{60}}{D_{10}} C_{c} = \frac{D_{30}^{2}}{D_{10}D_{60}}$$
(8)

5.3.2.6 Constant Hydraulic Head Conductivity Test Method

Constant head tests are generally used for sands that contain little silt or fines. A permeameter or hydraulic conductivity cell is used when the soil specimen is compacted or placed. Water flows from a tank through a column of soil (permeameter) which remains under constant head. As stated in the previous chapter, hydraulic conductivity measures the flow rate of water through a soil column. The standard, ASTM D2434-68, was used in all hydraulic conductivity testing and is presented in simplified form in Table 5-7. The standard was limited to disturbed granular soil containing not more than 10% soil passing the 75 mm sieve. The following are prerequisites for laminar flow and constant head conditions: continuity of flow, no soil volume change during test, soil voids saturated with water and flow is steady state with no changes in hydraulic gradient.

*	Procedure					
1	Prepare system which includes the CONStant head reservoir. all joints and tubing					
2	Prepare cell or permeameter					
3	Measure internal permeameter dimensions (diameter, length, etc.)					
4	Select representative test specimen that has been air dried					
5	Assemble bottom part of permeameter (cap, geomesh and geotextile)					
6	Place test specimen into permeameter according to dry pouring method					
7	Use funnel to pour specimen in a spiral motion					
8	Measure length of specimen					
9	Assemble top part of permeameter (cap and geotextile)					
10	Connect all tubing and check joints (inlet/outlet)					
11	Apply vacuum and saturate specimen					
12	Measure length of specimen again					
13	Remove vacuum and begin downward flow of water					
14	Measure change of head and flow when steady state is reached					
15	Repeat test at another change of head					
16	Dismantle permeameter					

Table	5-7:	Simplified	Constant-head	Hydraulic	Conductivity	Procedures.
				•/	•/	

Certain procedures from the standard have been omitted or altered slightly. The

constant head reservoir used tap water and was not deaerated. The specimen was placed in the permeameter with a funnel in three lifts. After each lift, the specimen was compacted with a rod. Note there was no specific relative density required. Also, instead of using porous stones, geotextiles and geomeshes were used to prevent specimen migration. Finally, there was no vacuum applied to the system. During saturation, a large constant head was applied and the permeameter was allowed to bleed off air.

5.3.3 Material Identification

5.3.3.7 Hydraulic Conductivity System

The system consists of two main parts, the permeameter and the constant head reservoir. The permeameters were constructed using schedule 40 PVC pipe with an inside diameter of 4 in (10.2 cm) and total length of 12 in (30.5 cm) (Figure 5-12).



Note: Figure is exaggerated to show detail.

experiments.

The bottom end of the permeameter was permanently capped with a thin plastic test cap and the other end consisted of a removable cap to allow soil specimen introduction and chemical additives. Inlet/outlets were threaded brass fittings that allowed quick disconnect and attachment to tubing for fluid transfer. Additional system equipment consisted of geotextile material and geomesh provided by GSE (formerly Gundle Inc.) to act as upper and lower screens to prevent soil movement. The constant head reservoir was fabricated from a plastic tank. Utilizing parts from a toilet repair kit, the tank was connected to a sink faucet to maintain constant hydraulic head.

5.3.3.8 Chemicals

In the factorial experiment design, calcium chloride $(CaCl_2)$, sodium carbonate (Na_2CO_3) and diammonium sulfate $((NH_4)_2SO_4)$ were used (Table 5-8). All chemicals had the purity grade of purum, which indicates it was at least 97% pure. These chemicals were observed to have some deviation from expected color, but not overly dramatic.

Chemical Formula Form Quantity Crystal lumps CaCl₂ calcium chloride (anhydrous) 6 kg (NH4)2SO4 Granular Ammonium sulfate 5 kg Sodium carbonate (anhydrous) Na₂CO₃ Granular 5 kg

Table 5-8: Chemical Formula, Form and Quantity.

To calculate the amount of chemical needed in all experiments, the anticipated volumes of calcium carbonate (CaCO₃) and calcium sulfate (CaSO₄) precipitates were first set equal to each other. This was done to make sure no particular chemical would have an advantage of reducing the hydraulic conductivity of a soil specimen based solely on amount of precipitate deposited. A sixteen cubic centimeter volume of anticipated precipitate was chosen for the experiments, which took into account the permeameter and specimen void space. The CaCl₂, Na₂CO₃ and (NH₄)₂SO₄ solutions were near saturation with molar concentrations of 4.33 M (327.2 g/l), 0.59 M (62.91 g/l) and 3.46 M (457.7 g/l) respectively. The reactants required for a CaCO₃ application were 100 ml of CaCl₂ and 730 ml of Na₂CO₃, and for a CaSO₄ application were 80 ml of CaCl₂ and 100 ml of (NH₄)₂SO₄.

5.3.3.9 Experiment Soils

The factor of initial hydraulic conductivity was divided into two levels, 10^{-2} or $10^{-3.5}$ cm/s. First, the base material, silica sand, was classified by performing a particle size analysis and constant head hydraulic conductivity test. One hundred pound bags of silica sand were used. It was classified



Figure 5-13. Grain size analysis of silica sand

as SP (USCS) or poorly graded sand and was a 40/60 mix, which indicates that most of the particles were held in the #40 (425 mm) and #60 (0.25 mm) sieves (Figure 5-13).

The next step was the addition of a certain percentage of bentonite clay to the base material to make different soil specimens. The clay was standard western bentonite bought from Bentonite Corporation, Denver CO. Percentage of bentonite clay in a soil specimen was calculated by volume. For instance, if the specimen were to have 10% clay, there would be nine measuring units of silica sand per one unit of bentonite clay,

Soil Specimen	Hydraulic Conductivity (cm/s)
16.7%	1.48E-06
13.0%	7.90E-06
12.0%	1.84E-05
10.0%*	3.62E-05
7,7%	1.65E-04
6.9%*	2.18E-04
6.3%*	1.29E-03
5.0%*	6.82E-04
0.0%*	2.09E-02

Table 5-9: Hydraulic Conductivity Values for Soil Specimens.

*Multiple specimens run

At least twenty soil specimens were made for hydraulic conductivity testing (Table 5-9). In Figure 5-14, there are two lines referred to as Soil 1 and Soil 2. These lines represent soils with a hydraulic conductivity of 10^{-2} cm/s and $10^{-3.5}$ cm/s, respectively.



Figure 5-14. Initial permeability of silica sand with added fraction of bentonite

Also, a soil specimen tit line was added to better locate which soil specimen crossed near the Soil 2 line. Soil 1 was later chosen to be a soil specimen containing no bentonite. A soil specimen, which contained 6.9% bentonite clay, was chosen as Soil 2.

53.4 Experimental Design

5.3.4.10 Experimental Setup

As discussed earlier a total of 16 experiments were performed. Each experiment began with permeameter preparation, which included a washing process. This process started with washing the entire permeameter with detergent, rinsing with tap water, rinsing with 10% muratic acid (HCl), and then rinsing again with tap water.

Another part of preparation was making sure the bottom cap was watertight. Water was either placed in a permeameter or connected to a constant head reservoir and left for several hours. If water was observed leaking from the system, a new cap was placed on the bottom or silicon was reapplied.

Once the system was watertight and clean, the permeameter was ready for soil introduction (Soil 1 or Soil 2). Eight geomeshes (large plastic screen) were put into the permeameter first to lift the soil specimen up from the bottom outlet approximately 2 in

(5.0 cm). Next, two geotextiles (fine fabric screen) were placed into the permeameter. These materials prevented soil from escaping or exiting the permeameter through the outlet.

Next, Soil 1 or Soil 2 was added according to the hydraulic conductivity test procedure. In each experiment, the length of specimen was approximately 6 in (15 cm), which left space for precipitate deposition.

After chemical application, another geotextile was placed into the permeameter on top of any deposits. The geotextile prevented the constant head reservoir water from eroding any deposits by distributing the water.

Finally, the removable cap was coated with vacuum grease and placed on the permeameter. Vacuum grease was also added to all threaded fittings to prevent air from leaking into the system. With these conditions met, the permeameter was ready to be connected to the constant head reservoir and begin the hydraulic conductivity test, which varied in length from 1 to 3 weeks in duration.

5.3.4.11 Chemical Application

The two chemical application techniques used in the laboratory are referred to as combined and alternating. Chemical solutions were prepared using the previously described methods. Tables 5-10 and 5-11 have been provided describing procedures for $CaCO_3$ and $CaSO_4$ precipitates. Applications were performed every twelve hours until 5 or 10 applications had been accomplished. Also, the temperature and the pH of the chemical solution and constant head reservoir were monitored and recorded.

5.3.4.12 Soil Specimen Evaluation

Once the final hydraulic conductivity has been measured for a particular experiment, the soil specimen was extracted from the permeameter. Usually, the specimen would slide out easily but sometimes air was added push the specimen out. The specimen characteristics were observed, noting items such as color of soil and amount of remaining deposit, Some specimens were also dissected to note location of precipitate deposition. Figures 5-15 through 5-17 illustrate the results of the sealing experiments.

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	Combined	Alternating
1	Prepare permeameter.	Prepare permeameter.
2	Measure 730 ml of 0.59 M Na ₂ CO ₃ solution in graduated cylinder.	Measure 730 ml of 0.59 M Na ₂ CO ₃ solution in graduated cylinder.
3	Measure 100 ml of 4.33 M CaCl ₂ solution in graduated cylinder.	Measure 100 ml of 4.33 M CaClz solution in graduated cylinder.
4	Record temperature and pH of each solution.	Record temperature and pH of each solution.
5	Pour Na2CO3 solution into large beaker and stir.	Pour Na ₂ CO ₃ solution slowly into open permeameter, trying not to disturb soil or previous application.
6	Slowly add CaCi2 solution to stirred solution.	Allow Na ₂ CO ₃ solution to pass below the surface of the soil.
7	Stir for approximately 2 minutes or until floc has formed	Slowly add CaCl ₂ solution to permeameter.
8	Pour combined solution slowly into open permeameter, trying not to disturb soil or previous annlication	Repeat steps 1-7 until desired number of applications has been reached.
9	Repeat steps 1-8 until desired number of applications has been reached.	

Table 5-10: Chemical Application Techniques for $CaCO_3$ Precipitate.

Table 5-11: Chemical Application Techniques for CaSO₄ Precipitate.

Ħ	Combined	Alternating
1	Prepare permeameter.	Prepare permeameter.
2	Measure 100 ml of 3.46 M (NH4)≥SO4 solution in graduated cylinder.	Measure 100 ml of 3.46 M (NH ₄) ₂ SO ₄ solution in graduated cylinder.
3	Measure 80 ml of 4.33 M CaCl₂ solution in graduated cylinder.	Measure 80 ml of 4.33 M CaCl ₂ solution in graduated cylinder.
4	Record temperature and pH of each solution.	Record temperature and pH of each solution.
5	Pour (NH4)2SO4 solution into large beaker and stir.	Pour (NH4)2SO4 solution slowly into open permeameter, trying not to disturb soil or previous application.
6	Slowly add CaCl ₂ solution to stirred solution.	Allow (NH4)2SO4 solution to pass below the surface of the soil.
7	Stir for approximately 2 minutes or until floc has formed.	Slowly add CaCl ₂ solution to permeameter.
8	Pour combined solution slowly into open permeameter, trying not to disturb soil or previous apolication.	Repeat steps 1-7 until desired number of applications has been reached.
9	Repeat steps 1-8 until desired number of applications has been reached.	



Figure 5-15. Photographs of experiment No. 1



Figure 5-16. Photographs of experiment No. 2.



Figure 5-17. Photographs of experiment No. 13 with calcium sulfate

5.3.5 Results & Discussion of Self-sealing Experiments

Experimental data were analyzed using the software *JMP*. The first model fit the experimental data to a linear model by least squares. This model focused attention to which factors were significant by comparing the sum of squared residuals to the sum of

squared residuals of the model with that factor removed. When shown graphically, leverage plots indicate what the residual was with and without that factor in the model. Also shown on the leverage plots are confidence curves. The confidence curves helped indicate whether the experiment was significant at a 95% level and whether or not a factor was significant. If the confidence curves cross the horizontal line, the factor was significant. If the confidence curves do not cross the horizontal line, the factor was not significant. Confidence curves that were asymptotic to the horizontal line were borderline and may or may not be significant.

The second model analyzed the experimental data with a screening process. The Screening Model can be used to analyze data where there are many factors and few observations. Traditionally, a screening model is used to optimize an existing model but it has been used here to graphically show significant factors in prediction profile and interaction plots.

The goal of the experiments was to seal or reduce the hydraulic conductivity of the soil. In the laboratory, there was reduction of the hydraulic conductivity in most experiments but only one achieved the desired permeability of 10⁻⁷ cm/s, which is the requirement for most clay liner systems. In Table 5-12, the final hydraulic conductivity for each experiment is presented with their respective test factors. Also, the final column labeled factor change, indicates the order of magnitude change from the initial hydraulic conductivity to the final hydraulic conductivity. For example, a value of 10 would represent 1 order of magnitude reduction of the hydraulic conductivity.

In experiments 1 through 8, with an initial hydraulic conductivity of 2.09 E-02 cm/s, final hydraulic conductivity decreased less than an order of magnitude. Experiments 9 through 16 again used a soil with 6.90% bentonite clay and an initial hydraulic conductivity of 2.18 E-04 cm/s. At least an order of magnitude decrease was observed for experiments with CaCO₃ precipitate; this included experiments 9, 11, 14 and 15. Experiments 10, 12 and 13 with CaSO₄ precipitate had a slight increase in final hydraulic conductivity and therefore did not exhibit any self-sealing characteristics (reduced hydraulic conductivity).

*	Chemical Solution	Chemical Application Technique	Number of Applications	Initial Hydraulic Conductivity (cm/s)	Final Hydraulic Conductivity (cm/s)	Factor Change
1	CaSO₄	Combined	5	2.09E-02	1.39E-02	1.5
2	CaCO ₃	Combined	10	2.09E-02	3.20E-03	6.5
3	CaCO₃	Combined	5	2.09E-02	1.04E-02	2.0
4	CaSO4	Alternating	10	2.09E-02	1.12E-02	1.9
5	CaSO4	Combined	10	2.09E-02	9.83E-03	2.1
6	CaCO ₃	Alternating	5	2.09E-02	5.49E-03	3.8
7	CaCO ₃	Alternating	10	2.09E-02	4.06 E-0 3	5.1
8	CaSO ₄	Alternating	5	2.09E-02	1.55E-02	1.3
9	CaCOs	Combined	10	2.18E-04	1.82E-05	12.0
10	CaSO4	Alternating	5	2.18E-04	1.94E-03	0.1
11	CaCO ₃	Alternating	5	2.18E-04	1.41E-05	15.5
12	CaSO4	Combined	5	2.18E-04	9.46E-04	0.2
13	CaSO4	Alternating	10	2.18E-04	8.44E-04	0.3
14	CaCOa	Combined	5	2.18E-04	2.84E-07	768.7
15	CaCO ₃	Alternating	10	2.18E-04	1.69E-06	128.8
16	CaSO4	Combined	10	2.18E-04	6.53E-05	3.3

Table 5-12: Final Hydraulic Conductivity and Factors.

5.3.5.13 Least Squares Model

First, each test factor (X) was assigned a modeling type of continuous, ordinal or nominal, Continuous data is numeric in nature and is used directly in the model. Ordinal data can be either numeric or character but is interpreted as having discrete values or ordered categories. Nominal data can either be numeric or character but is treated only as unordered discrete values. All test factors were treated as nominal data for several reasons. Each test factor was either at one level or another. For instance, the number of applications factor was either 5 or 10 applications and nothing in between. In contrast, the response value (Y or log(Initial k /Final k)) was designated to be a continuous model type. The log(Initial k / Final k) was used in modeling for several reasons. First, the hydraulic conductivity was a numeric calculated value and was expected to have some random component. Next, the logarithm of Initial k over Final k normalized the response value and also revealed which experiments had a considerable reduction in hydraulic conductivity.

JMP can fit many models such as multiple regression, models with complex effects and multivariate models. Because the response value was continuous, the fitting model was a specific form (Eqn. 9), where log (Initial k / Final k) equals the logarithm of the initial hydraulic conductivity over the final hydraulic conductivity, **S** refers to the different soils, C refers to the different chemicals, A refers to different application techniques, and N refers to the number of applications. This form assumed that error is normally distributed.

$$Y = [F(X's) \text{ and parameters}] + error$$

$$\log\left(\frac{initialK}{finalK}\right) = (S, C, A, N) + error$$
(9)

The fitting principle for both the Least Squares Model and the Screening Model was the standard least squares method. This method minimizes the sum of squared errors between the actual response value and the predicted values. The analysis estimates the model that gives the most likely residuals normally distributed.

Since all test factors were nominal and a linear model was utilized, the model form had levels with predicted coefficients. The predicted coefficients represent differences from each level to the average response across all nominal values. For instance, suppose a nominal factor called Color of Soil has three levels: red, green and black (JMP Statistics and Graphics Guide, 1995). The model will have an intercept and two parameters that show a value to add to the intercept if the level of Color of Soil is red or green (Table 5-13). When Color of Soil factor is black, you subtract both Color of Soil parameters from the intercept to get the predicted value.

Term Estimate						
B0	intercept	7.9179				
B1	Color of Soil (red)	-1.2054				
B2	Color of Soil (green)	0.0702				

Table S-13: Intercept and Parameter Estimates for Example Color of Soil (Green).

The equation would have the following linear form,

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + error \tag{10}$$

where β_0 equals the intercept, β_1 and β_2 are parameters for Color of Soil (red) and (green),

$$7.9179 + \left\{ \begin{cases} 1.1352, & \text{if Color of Soil} = black \\ 0.0702, & \text{if Color of Soil} = green \\ -1.2054, & \text{if Co/or of Soil} = red \\ ?, & \text{otherwise} \end{cases} \right\}$$
(11)

The actual prediction formula for Y produced by JMP does not look exactly like (Eqn. 10) but rather appears in the following form (Eqn. 11). The form of (Eqn. 11) resembles the predicted equation for log(Initial k / Final k) except it has ten levels (Eqn. 12).

The final predicted equation fit the experimental data well. In Table 5.14, the \mathbb{R}^2 estimated the proportion of the variation in the lo&Initial k / Final k) around the mean that can be attributed to test factors in the model rather than random error. A value of 1 would represent a perfect fit. A value of approximately 0.83 indicated an adequate fit for the experimental data.

Table 5-14: Summary of Fit for Predicted Equation.

Parameter	Value	
R²	0.832429	
RAdjusted	0.497288	
Root mean square error	0.683712	
Mean of response	0.55417	
Observations	16	

It is meaningful to remember that the final equation was not necessarily the most



(12)

important aspect of the analysis. The leverage plots provided in the least squares model and the interaction plots provided in the screening model were also important because they facilitated locating significant test factors.

There were two types of leverage plots, the Whole Model leverage plot and the Effect in Model leverage plot (Figures 5- 18 and 5-19). The whole model leverage plot shows the experimental response versus the predicted response. This leverage plot shows a point by point display of how the hypothesis sum of squares was composed. The Whole Model hypothesis represents all the parameters except



Figure 5-18. Whole model leverage plot.

the zero intercept without any effects in the model. The distance from a point to the 45° line shows the actual residual. The distance from the point to the horizontal line at the mean shows what the residual error would be if all effects were removed from the model (JMP Statistics and Graphics Guide, 1995).

The Effect in Model leverage plot shows a point by point display of how the hypothesis sum of squares was composed. The distance from a point to the regression line was the actual residual. The distance from a point to the mean without that particular effect shows what the residual error would be if the effect were removed from the model (JMP Statistics and Graphics Guide, 1995).

Reiterating, the dashed curves in the leverage plots represent 95% confidence level lines. If confidence level lines cross the horizontal line (mean) then the factor is significant. If the confidence level lines do not cross the horizontal line then the factor is not significant. If the confidence level lines approach the horizontal line but do not



Figure 5-19. Effect in model leverage plot.

necessarily cross it then the factor is called borderline. It may or may not be significant.

In the experiment Whole-Model leverage plot (Figure 5-20), measured $\log(\text{Initial k / Final k})$ and predicted $\log(\text{Initial k/Final k})$ values were shown with the 45° line and 95% confidence curves. Note the confidence curves appear on either side of the regression line and experiments appear as individual dots. This plot indicated that all factors together did not explain a major proportion of the variation in the lo&Initial k /Final k). The next step was to look at the four individual factors and interaction leverage plots.



horizontal line of *Figure 5-20*. Leverage plot of whole model results. their respective

plots. The other factors, Application Technique, Number of Applications and Initial k (cm/s), were determined not to be significant factors since their confidence curves did not cross the horizontal.

In Figure 5-22, interaction leverage plots to the second degree are presented. The only leverage plot that showed possible significance was Chemical * Initial k (cm/s). Again, the confidence curves approached the horizontal line and therefore it is borderline. All other interaction leverage plots exhibit confidence curves that do not cross the horizontal line or even approach asymptotic behavior. This indicates that these factor interactions were not significant to the response value, log(Initial k / Final k).



Figure 5-21. Single effect leverage plots

Besides looking at the leverage plots to determine factor significance, one can also examine the t-ratio or F-ratio values calculated during the JMP analysis (Table 5-15). The t-ratio is formed by finding the difference between the factor estimate and the hypothesized value (equal to zero) and then dividing that quantity by its standard error (JMP Statistics and Graphics Guide, 1995). If the t statistic is greater than an absolute value of 2 and the probability oft (Prob>t) is less than 0.05 then the factor is significant. The F-ratio is the mean square of the model divided by the mean square of the error. The F-ratio is a measure of improvement in fit when separate means are considered. If the F-ratio is quite large and the probability of F (Prob>F) is less than 0.05 then the factor is significant. In Table 5-15, the t and F test values concur with the graphical results.

Term	Estimate	Std Error	t quico	Prob > t	Fato	Prob > F
Intercept	0.554	0.170	3.24	0.022	NA	NA
Chemical	0.647	0.170	3.79	0.012	14.351	0.0128
Application	-0.131	0.170	-0.77	0.477	0.590	0.4770
Chemical* Application	0.060	0.170	0.35	0.739	0.123	0.7395
Number	-0.093	0.170	-0.55	0.608	0.298	0.6081
Chemical* Number	0.133	0.170	0.78	0.470	0.610	0.4700
Application*Number	-0.091	0.170	-0.53	0.616	0.284	0.6164
Initial k (cm/s)	-0.140	0.170	-0.82	0.449	0.672	0.4495
Chemical' Initial k (cm/s)	-0.459	0.170	-2.69	0.043	7.217	0.0435
Application*Initial K (cm/s)	0.139	0.170	0.82	0.450	cl.670	0.4501
Number*Initial	-0.023	0.170	-0.14	0.897	0.018	0.8972

Table S-15: Summary oft and F Test Whole Model.

From the Least Squares Model, all test factors (Chemical Solutions, Application Technique, Number of Applications and Initial Hydraulic Conductivity) exhibited little importance to the predicted model and therefore did not necessarily effect the final hydraulic conductivity. By looking at each factor individually, one factor, Chemical Solutions, appeared to be more significant than the others. In addition, the interaction of Chemical Solutions and Initial k (cm/s) was also observed to be significant in reducing the final hydraulic conductivity. All other individual factors and interactions were not significant.

5.3.5.14 Screening Model

Usually, a screening model is used to "screen" test factors when there are many effects and few observations. Although this model does not produce any leverage, residual plots or least squares statistics, it does provide prediction and interaction profile plots. Prediction profile plots graph the least squares means where one factor forms the X axis and the final hydraulic conductivity forms the Y axis. Interaction plots show the scale of interaction by graphing the predicted values of combinations of two test factors while holding the rest constant. For example, in interaction plots, if the lines plotted were parallel, there would be no factor interaction. The clarity in the interaction plots enabled easier identification of significant test factors. In Figure 5-23, the interaction profile plots illustrate the significant test factors a different way. The plot graphs each test factor in a matrix form. When a factor was moved in the prediction profile in JMP, each factor would change according to the model and this in turn showed the degree of interaction between factors. In Figure 5-23, there are two



Figure 5-22. Interaction leverage plots



Figure 5-23. Interaction profile plots.

lines in each block. These lines correspond to a particular factor. For instance, in the lower left-hand comer of the figure, the block contains the two lines. Each line represents a different initial hydraulic conductivity, Soil 1 and Soil 2. This factor is then compared to the chemical solutions, $CaCO_3$ and $CaSO_4$. Since the lines are not parallel in this particular block, this means that there is an interaction between the chemical solutions and initial hydraulic conductivity. Also from this block, $CaCO_3$ appeared to reduce the hydraulic conductivity of the soil better than $CaSO_4$.

Another insight from the interaction plots was that neither Application Technique nor Number of Applications test factors had any major effect on the reduction of the final hydraulic conductivity. In the second column of blocks, Application Technique vs. the other three test factors, all lines were approximately parallel and nearly horizontal. This meant that if you used either application technique you would produce similar results. In the third column of blocks, Number of Applications vs. the other three test factors, the lines were also approximately parallel and horizontal. This factor was also designated as not significant because little variation was observed when test factors were modified in the prediction profile.

In the Screening Model, the same conclusions as the Least Squares Model can be made: Chemical Solutions was significant and Application Technique, Number of Applications and Initial k (cm/s) were not significant. Furthermore in Figure 5-23, the Chemical Solutions and Initial k (cm/s) plot also exhibited interaction.

5.3.5.15 Soil Specimen and Analysis Evaluation

As stated previously, experiments 1 through 8 used Soil 1 with an initial hydraulic conductivity of 2.09 E-02 cm/s (Table 5.12). Experiments 1 through 8 had a reduction in hydraulic conductivity of less than one order of magnitude. Also, $CaCO_3$ generally created a lower hydraulic conductivity than $CaSO_4$. This reinforces the results analyzed in *JMP* that the use of $CaCO_3$ would reduce the hydraulic conductivity of a soil better than $CaSO_4$.

Furthermore, when evaluating the extracted specimens, $CaCO_3$ maintained its initial size after chemical application and hydraulic conductivity testing. It appeared as though dissolution *or* relocation of $CaSO_4$ into the soil voids might have occurred. In Figures 5-16 and 5-17, Experiment 2 ($CaCO_3$) and Experiment 13 ($CaSO_4$) are presented. In both photographs, a white layer appears on the left. In Experiment 2, the $CaCO_3$ layer was 1.5 *in*. (3.8 cm), which was approximately the same thickness prior to hydraulic conductivity testing. In Experiment 13, the $CaSO_4$ layer was 0.25 in. (0.6 cm), which differed from the original 0.75 in. (1.9 cm) prior to hydraulic conductivity testing.

In experiments 9 through 16, the initial hydraulic conductivity was 2.18 E-04 cm/s. There was at least an order of magnitude reduction in hydraulic conductivity in all experiments with the $CaCO_3$ solution. Some experiments utilizing $CaSO_4$ had a slight increase in permeability. This might indicate that, similar to the previous experiments, the $CaSO_4$ was going into solution during the hydraulic conductivity testing and was either washed out of the permeameter or moved deeper into the soil voids.

5.3.5.16 Further Analysis of Chemical Solution and Initial Hydraulic Conductivity Interaction

In view of the possible dissolution or relocation of $CaSO_4$ precipitate and a slight increase in hydraulic conductivity measured in experiments 10, 12 and 13, further analysis was performed on the interaction of Chemical Solutions and Initial k (cm/s). The analysis consisted of separating the data into $CaCO_3$ experiments and $CaSO_4$ experiments. For each set of experiments, a linear fit was again utilized in the JMP software, where the log(Initial k/Final k) was the response value (Y) and Initial k (cm/s) was the factor (X). In addition, t and F tests were also calculated to determine significance. Separating the experiments by their respective precipitate allowed conclusions to be drawn on whether the chemical affected the final hydraulic conductivity of the soil.

Several findings were detected in the analysis of the $CaCO_3$ experiments. First, experiments 2, 3, 6, and 7 using Soil 1 had little variation in their final hydraulic conductivity. Next, experiments 9, 11, 14 and 15 using Soil 2 had notable variance in their final hydraulic conductivity. Experiments 14 and 15 had much lower hydraulic conductivity of 10^{-6} to 10^{-7} . t and F tests for the CaCO₃ experiments also illustrated the significance of the Initial k (cm/s) factor (Table 5.15).

For the $CaSO_4$ experiments the analysis also concluded that Soil 1 (experiments 1, 4, 5, and 8) had little variation in final hydraulic conductivity and Soil 2 (experiments 10, 12, 13, and 16) had great variation in final hydraulic conductivity. However, t and F tests for the $CaSO_4$ experiments indicated that there is no significance of the Initial k (cm/s) factor (Table 5.15).

One interpretation for significance of Initial k (cm/s) in $CaCO_3$ experiments and lack of significance in the $CaSO_4$ experiments is that $CaSO_4$ may have affected the bentonite clay mixed in Soil 2 by causing flocculation.
5.4 Self-Sealing Experiments in the Field

Four field permeameters, shown in Figure 5-24, were constructed of steel cylinders underlaid by a coarse sorted gravel to facilitate drainage. The water eventually drains out of the gravel into an adjacent salt marsh.

Field treatments were made by mixing ion exchange waste regenerate with sodium carbonate



Figure 5-24. Field permeameters.

forming a milky white precipitate of calcium carbonate. The solution was mixed in a plastic trash can then pumped and sprayed on the soil surface. A variety of treatments were tried including a) seeding the soil with sodium carbonate followed by watering b) spraying the precipitate on the top of the soil surface, c) alternating layers of treatment with addition of more sand, and d) mixing bentonite with the precipitate and spraying the resultant mixture. Photographs of spray application and a dried layer of precipitate are shown in Figure 5-25.



Figure 5-25. Application of sealing solution and view of the bottom of an infiltration testing ring after drying.

Results are shown in Figures 5-26 to 5-28. Permeameter 2 initially was treated with calcium carbonate precipitate on the surface. This was followed by the addition of sand layers

and precipitate along





with limited compaction. The treatments reduced permeability from 0.016 cm/s to 0.0005 cm/s – more than two orders of magnitude. However, the lowest permeability obtained was well above the desired level of 10^{-7} cm/s.



Figure 5-27. Response of permeameter 3.

volumes of

included equal

bentonite and sodium carbonate. Results for permeameter 3 are shown in Figure 5-27.

Permeameter number 4 (Figure 5-28) was initially filled with mixed sand and bentonite giving a lower starting permeability. Precipitate was sprayed on the surface, further lowering permeability. In conclusion, all the field experiments reduced field permeability significantly, but not to the extent desired. Further laboratory tests are underway to determine optimal treatment methods.

5.5 Enhanced Evaporation Experiment

After several design revisions, the enhanced evaporation tests were ready and underway by February/March 1998. Bay 1 and Bay 2 were employed during the tests. Bay 1 was fitted with four spraying towers, and Bay 2 was used

as a control to measure



Figure 5-28. Response of permeameter 4.

natural evaporation. The PDP building was fitted with a weather station connected to a computer to collect the following data: outside temperature, relative humidity, solar radiation, precipitation, wind speed and direction, and barometric pressure.

The spraying towers were constructed of 1.5 ft (0.46 m) irrigation control valve boxes. Stainless steel spray heads were connected on top of each tower. Two types of spray heads were used: a wide-spread, low-profile spray head and a narrow-spread, high-profile spray head. Two $\frac{1}{2}$ hp sump pumps were placed in a plastic box at the deep end of the pond with straining nets to remove any suspended solids from the concentrate as it is pumped to the spray head. The straining nets were used to prevent any clogging of the spray heads. Each sump pump supplied concentrate to the two (2) towers,

Figure 5-29 shows the cumulative evaporation loss between the two bays. Bay 1 shows a fast loss over Bay 2 at the start of the experiment, but as the bays become more concentrated the net evaporation rate becomes steady. The vapor pressure of the concentrate decreases as dissolved solids content increases. As more water was evaporated, regardless of increased surface area provided by sprayers, the rate of evaporation between the bays was not significantly different over the short study period.



Figure 5-29. Change in evaporation: sprayers versus control bay.



Figure 5.30. Concentration profile: sprayers versus control bay.

5.6 Salt-Tolerant Plants

In October 1997, a small lined pond (10 x lo-foot) filled with native soil was constructed. Cattails were harvested from San Elizario, Texas in El Paso County where they grow wild along irrigation ditches and the sides of irrigated fields. The cattails were then transplanted to the pond and watered with raw feed water (1,100 to 1,300 mg/l TDS). Of the eight bulbs that were transplanted only one survived through the winter months.

In March 1998, the surviving cattail was transplanted to the sump area where native plants and animals were thriving. Saline water from the ion exchange regeneration wastewater was introduced periodically to keep the sump (or lagoon) water saline. The table following lists the quality of the water used for irrigating the cattails. Since the second transplant, the surviving cattail has produced four sprouts and continues to grow.

Chemical Parameter	Value
TDS	14,000 mg/l
Silica	not recorded
Total Alkalinity (average)	58 mg/l as CaCO,
Total Hardness (average)	1,750 mg/ as CaCO,

Table 5-16: Characteristics of Wetland Irrigation Water.

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