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TECHNICAL REPORT:

XYZ Company

COMPILED FOR:

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INNOVATIONS IN ION EXCHANGE

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INTRODUCTION

XYZ Company is looking for a way to remove greater than 90% of the target species in their organic solution. ResinTech was tasked with the removal of these inorganic reactants from this aqueous organic solution. The experiment was run in a packed bed, up flow configuration to keep the anion resin from floating during the service cycle.

Appendix B contains the comprehensive Project Experimental Outline

OBJECTIVE

The objective is to determine the throughput capacity and leakage for three target species (sodium phosphate, manganese sulfate, and copper sulfate) for a separate bed demineralizer configuration, with hydrogen form cation (**CG8-H**) and weak base anion (**WBMP-OH**) polished with a Strong Acid / Strong Base mixed bed ion exchange resin, **MBD-15-SC**. The removal goal is <10% leakage of the targeted species.

EXECUTIVE SUMMARY

- Anion resin was confirmed to float in the XYZ Company Solution.
- The 2-Column configuration removed greater than 90% of the target species.
- Breakthrough started between 6.51 and 6.37 bed volumes as shown by the steep increase in conductivity and decrease in pH, respectively.
- Silica was the first ion to leak, followed by Phosphate then Sulfate and ultimately Chloride. Sulfate Leakage at 7.0 Bed Volumes was the first ion to exceed the objective.
- Sodium was the first target cation to leak, followed by Copper. Manganese was completely removed and did not leak.
- Dumping was exhibited of Silica, Sulfate, Sodium and Phosphate where the concentrations of these ions were present at concentrations greater than the influent upon exhaustion of the resin.
- The solution changed from clear to an amber color as the columns exhausted.

DATA ANALYSIS

All data is available in the Excel File: 080322 XYZ Company Data Final.xlsx. Below is an analysis of and excerpts from the laboratory data. The analysis below outlines multiple influent and effluent constituents and specifically the target items of interest to XYZ Company per the experimental objective. The data is overlaid with Ion Exchange Theory to explain what is happening in the columns and why.

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INFLUENT ANALYSIS

The organic solution provided was run on the ICP for cations. Some cations, Phosphorous and Silicon, are present in the solution as Anions. IC analysis was added at the end of the experiment to capture the Anion Concentrations. The target leakages are also defined in the table below in the Objective Column.

	mg/L	Influent Solution	Objective
	pH	NA	NA
	Conductivity	NA	NA
Cation Leakage	Sodium	631.20	63.12
	Copper	0.261	0.026
Anion Leakage	Silica	2.94	0.294
	Sulfate	13.78	1.38
	Phosphate	36.15	3.62
	Chloride	369.66	36.966
Cations with no Leakage	Zinc	0.147	0.015
	Magnesium	1.43	0.143
	Calcium	12.20	1.22
	Iron	3.43	0.343
	Manganese	0.471	0.047

EFFLUENT ANALYSIS

Effluent samples were collected and analyzed at various increments. The pH and Conductivity were recorded continuously, in line. The Exhaustion Curve plots the real time data of pH and Conductivity over the course of the run. The data collected is from the effluent of Column 2. No data was collected between Columns 1 and Column 2.

The Bed Volume Effluent Samples were collected in bulk and then analyzed. The effluent concentrations presented are the AVERAGE leakage concentrations over the bed volume range in question. A deep dive was done into all the data collected to better understand the entirety of what was seen during the purification of the solution. Initial analysis of ICP, Cation data and pH data showed the presence of Hydrogen (acid) appearing first in the effluent. The analysis was widened, and the IC was run to find the corresponding anions for the acid present.

The new data was tabulated into the 'The Comprehensive Leakage and Influent Solution Profile' table below, with bed volume analysis:

COMPREHENSIVE LEAKAGE PROFILE AND INFLUENT SOLUTION PROFILE

			Cations		Anions			
	pH	Conductivity	Sodium	Copper	Silica	Sulfate	Phosphate	Chloride
	SU	Micro Mho	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
Influent Solution	NA	NA	631.20	0.261	2.94	13.78	293.00	369.66
Bed Volume	6.06	0.196	5.37	x	x	x	x	x
0.0	6.06	0.196	5.37	x	x	x	x	x
0.7	6.00	0.177	4.74	x	x	x	x	x
1.4	5.76	0.671	0.74	x	2.89	x	x	x
2.8	5.64	0.673	x	x	4.61	x	7.41	x
4.2	5.58	0.865	x	x	7.66	1.00	22.5	x
5.6	5.50	1.106	x	x	4.70	0.77	32.8	x
7.0	4.13	13.209	x	x	7.38	6.00	14.4	x
8.4	4.29	154	1.36	x	27.18	102.70	396	x
9.8	4.54	588	4.41	x	17.23	56.71	341	x
11.2	4.79	1287	349.90	0.10	18.82	12.43	106	0.80
12.6	6.02	4249	1475.00	0.11	15.52	14.68	263	6.66
14.0	6.55	4294	1688.00	0.13	15.70	10.42	2112	81.21

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Bed Volume 0

the column starts up with some initial Sodium leakage that quickly rinses out. The product purification has started with the complete removal of all ionized constituents. The product is effectively 'DEMINERALIZED' and meets the Objective.

Bed Volume 1.4

Silica leakage has begun at 2.89 ppm. Silica is not a target ion per the stated objective but noted as exceeding the 10% leakage desired based on the influent analysis.

Bed Volume 2.8

Phosphate Leakage started at 7.41 ppm, below the 10% objective of 29.3 ppm.

Bed Volume 5.6

Phosphate Leakage was measure at 32.8 ppm, slightly above the 10% leakage objective of 29.3 ppm. The value reduces at the next measurement and the average leakage over bed volumes 2.8-7.0 remains in spec.

Bed Volume 7.0

Sulfate at 6.00 ppm and Phosphate at 396 ppm both exceed the objective leakages of 1.38 ppm and 29.3 ppm, respectively. The pH has dropped to 4.13, showing Silicic, phosphoric and Sulfuric Acid in the effluent. Silicic and Phosphoric acids are weakly ionized and not conductive. The presence of 6.00 ppm of Sulfate (sulfuric acid) is causing the drop in pH and increase in conductivity.

Bed Volume 8.4

1.36 ppm of Sodium is noted in the effluent, less than the 63.12 ppm maximum leakage. Sulfate and Silica leakage's have peaked and are now receding. At this point in the run, Carbon Dioxide and Bi-Carbonate must also be leaking from the strong base anion resin. The ratio of these ions will cause some buffering of the pH. The pH has started to rise to 4.29 but there is a hump in the curve that is most likely caused by the varying salt, acid and alkalinity mixture in the effluent. The leakage is now a combination of: Sodium Sulfate, Sodium Silicate, Silicic Acid and Sulfuric Acid. These Sodium salts and acids are very conductive as confirmed by a larger increase in conductivity.

Bed Volume 11.2

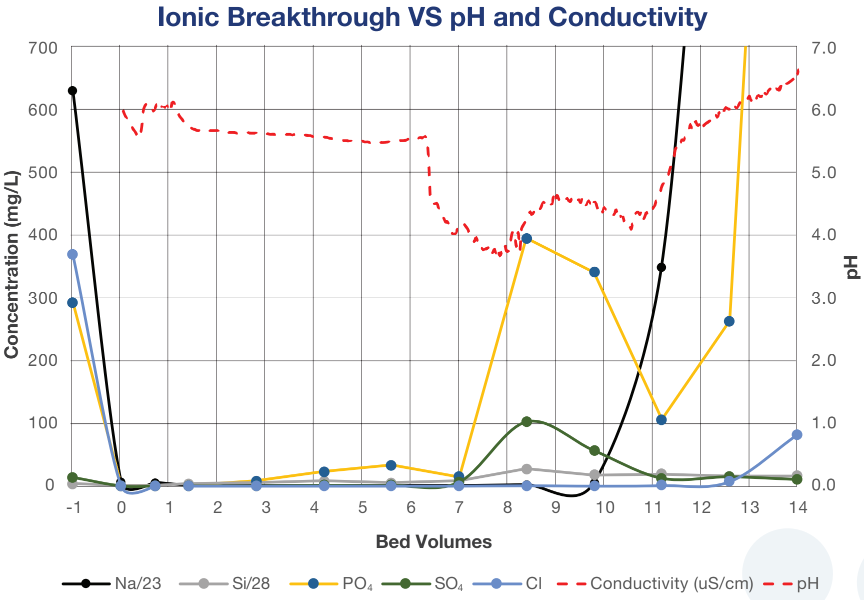
Copper has broken through the columns at 0.10 ppm. This exceeds the 10% target leakage defined in the objective. At this point we see breakthrough of every ion in the solution above the target thresholds. The run is over.

CATIONS THAT DO NOT LEAK:

The cations listed in the table below were found to be present in the influent organic solution. These cations were 100% removed during the purification process. No trace of these metals was found in the effluent analysis.

CATIONS THAT WERE REMOVED BUT DID NOT LEAK BY BED VOLUME

mg/L	Influent	0.0	0.7	1.4	2.8	4.2	5.6	7.0	8.4	9.8	11.2	12.6	14.0
Zinc	0.147	x	x	x	x	x	x	x	x	x	x	x	x
Magnesium	1.43	x	x	x	x	x	x	x	x	x	x	x	x
Calcium	12.20	x	x	x	x	x	x	x	x	x	x	x	x
Iron	3.43	x	x	x	x	x	x	x	x	x	x	x	x
Manganese	0.471	x	x	x	x	x	x	x	x	x	x	x	x



The ‘Ionic breakthrough versus pH and Conductivity’ graph shows distinct changes as the ions break through the column. It is critical to note that the pH and Conductivity were measured continuously while the ionic leakages are average values from a single sample collected over the entire bed volume. The data shown at -1 Bed Volume is the influent analysis of the solution provided by XYZ Company.

PH

The pH remains stable at around 6.0 until 6.37 bed volumes when it drops sharply, to 4.59. A drop in pH is typical of ionic leakage from a strong base anion resin. Silica and Carbon Dioxide are the least strongly held anions and the first to appear in the effluent. Carbon Dioxide leakage pH’s are typically above 5.3.

A drop in pH below 5.3 confirms the presence of free mineral acidity in the effluent. The indication is that **CG8-H** is converting cations to acids. The Anion Resins are nearing exhaustion with no hydroxide capacity left to exchange for the anions. Acids not removed by the anion resin appear in the effluent along with other anionic leakages. This is further supported by the rapid rise in conductivity at the same time.

The second pH inflection point is seen at 8.26 bed volumes. At this point, the pH starts to rise after dropping to a low of 3.68. At 10.57 bed volumes, the pH starts to increase rapidly. This is the suspected point where all resins are completely exhausted. Breakthrough of Sodium and Chloride occur.

CONDUCTIVITY

The mixed bed column is producing low conductivity, less than 2 micromho, (less than 1 ppm) for the first 6.475 bed volumes of the run. The rapid spike in conductivity indicates the exhaustion of one or more of the resins and the start of ionic slippage. Once breakthrough begins, the ions start to leave the resin bed in order of their selectivity and the conductivity will rise in steps as better seen in the Exhaustion Profile Graph, Appendix A.

SILICA

Silica is weakly ionized and the first anion to leak. It is measured on the ICP as Silicon but removed by the anion resin as Silica, SiO_2-. The spike in Silica is a dumping event and occurs because of Sodium Leakage from the cation resin. Sodium leakage in the cation resin causes a high pH, internal to the mixed bed and effectively regenerates the silica from the strong base anion resin. There is much more Sodium than Silica in the influent water. The higher concentration of Sodium Hydroxide is removed preferentially over the silica already on the bottom of the resin bed. The result is to quickly dump the silica removed by the bed in a very short period of time. This is evident by the short spike in silica at the same time as Sodium first appears in the effluent.

SODIUM

Sodium is the least strongly held of the cations. Sodium is the primary cation in the solution, more than 10 times the concentration that all other cations, combined. The Sodium also dumps off the resin beds in concentrations higher than the influent. The Sodium leakage appears higher than the combination of the influent Sodium and the sum of the other cations present. It seems that a component in the influent organic solution is ionized and removed more selectively, displacing the Sodium from the resin at the end of the run.

PHOSPHATE / PHOSPHOROUS

Phosphorous is measured as a cation on the ICP but present in the water as an anion, Phosphate (PO_4-). Subsequent analysis of the actual anions was done on the IC. The Phosphate measurements correlate more closely with traditional ion exchange theory which tells us the silica leakage would be followed by Phosphate, then Sulfate, and ultimately Chloride.

Phosphate was removed for 7.0 bed volumes while meeting the desired objective. Phosphate does appear to dump off the resin columns in concentrations greater than the influent once the Chloride starts to leak.

MANGANESE, COPPER, CALCIUM, MAGNESIUM, AND IRON

Strong acid cation exchange resins have a much higher affinity for divalent cations than monovalent cations. In order of selectivity, Copper is the least strongly held of these divalent ions. The Copper breaks through at 11.2 bed volumes, shortly after the Sodium breaks. The Copper does not appear to dump.

There is no presence of Iron, Calcium, Magnesium, or Manganese in the effluent. This agrees with traditional Ion Exchange operation and selectivity's.

CONCLUSION:

The resin configuration removed greater than 90% of the target species, less than 10% Leakage. Phosphate was the first target ion to leak but the average Phosphate is considered to meet the objective when looking at all the data. Sulfate was the first ion to exceed the target leakage at 7.0 Bed Volumes. The 2-column packed bed counter-current configuration treated 11.2 Bed Volumes to complete exhaustion.

The system is overall ANION LIMITED. Hydrogen was the first ion to appear in the effluent. An ion balance was done to compare Cations and Anions. The analysis seems to be missing a substantial quantity of Anions. Given the imbalanced analysis in conjunction with the dumping of the Sodium, it seems that some part of the XYZ Company Solution is ionized and uses resin capacity.

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Cations		mg/L (ppm)	mg/L as $CaCO_3$	meq/L
Sodium	as Na	631.0	1371.7	27.435
Potassium	as K	6.70	8.6	0.172
Magnesium	as Mg	1.43	5.9	0.118
Calcium	as Ca	12.2	30.5	0.610
			Sum of Cations	28.335

Anions		mg/L (ppm)	mg/L as CaCO3	meq/L
Fluoride	as F		0.0	0.000
Chloride	as Cl	369.7	521.4	10.428
Bicarbonate	as HCO_3	0	0.0	0.000
Nitrite	as NO_2		0.0	0.000
Bromide	as Br		0.0	0.000
Nitrate	as NO_3		0.0	0.000
Phosphate	as PO_4	293	462.7	9.255
Sulfate	as SO_4	13.8	14.4	0.287
			Sum of anions	19.969
			Ions Check RPD(%)	34.64

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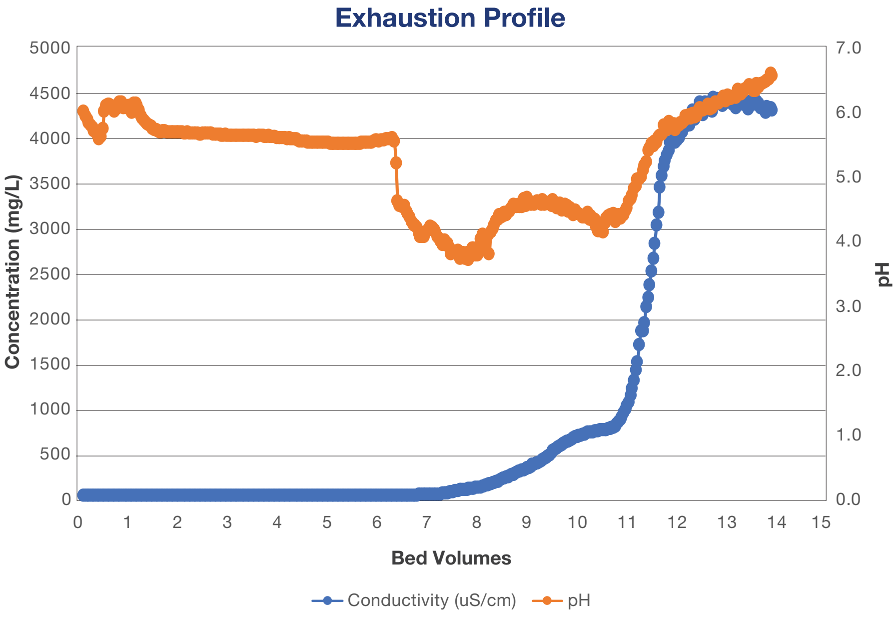
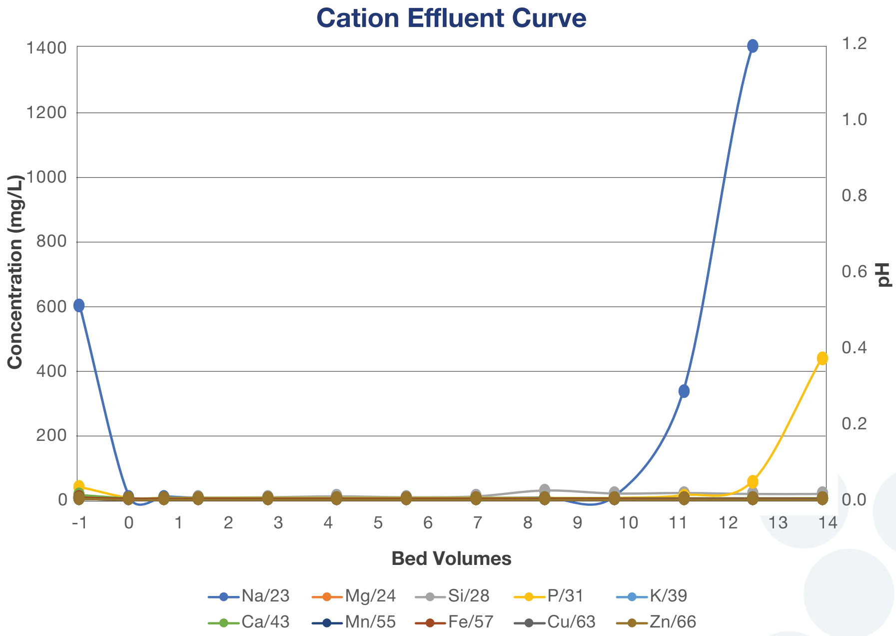
APPENDIX A: Data





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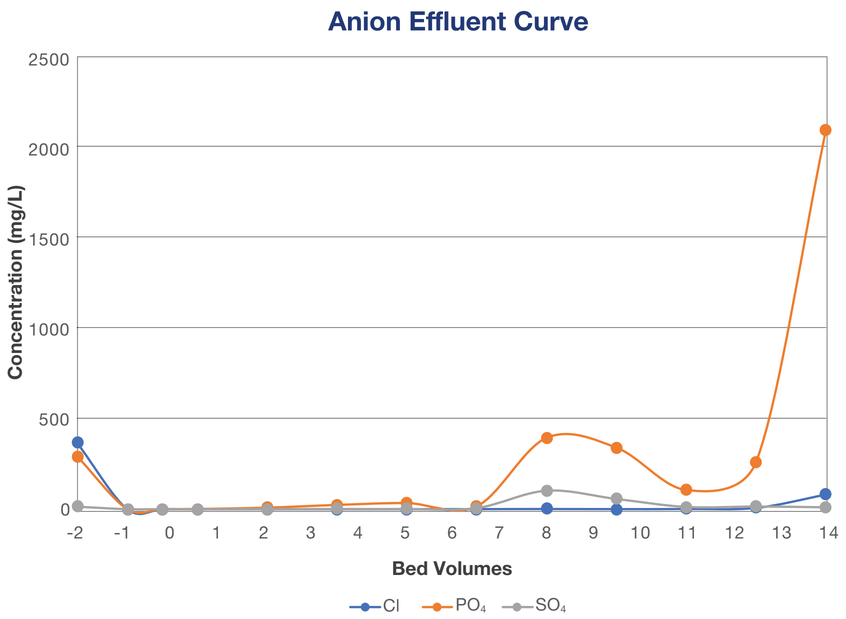
Anion Effluent Curve
Concentration (mg/L)
2500
2000
1500
1000
500
0
-2
-1
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
Bed Volumes
Cl
PO₄
SO₄

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APPENDIX B: Experimental Outline

Outline of proposed column work

OBJECTIVE

Removal of left-over inorganic reactants from aqueous organic solution. Determine the throughput capacity and leakage for three target species (sodium phosphate, manganese sulfate, and copper sulfate for a separate bed demineralizer configuration, with hydrogen form cation and weak base anion (**WBMP**). Provide basic process design including equipment sizes and chemical use sufficient for the client to estimate capital and operating costs and decide if ion exchange is economically viable

The removal goal is > 90% removal of all three targeted species.

SOLUTION CHARACTERIZATION

Matrix 200 gram per liter sugars
Specific gravity 1.1
Sodium mono and dibasic phosphate 15 mM
Manganese sulfate 50 uM
Copper sulfate 10 uM
pH around 7

BRIEF OUTLINE OF LAB WORK

Before running columns, analyze the solution sent to us. If it is not crystal clear, filter it until it is crystal clear. If possible, digest some of the suspended solids and analyze for the usual suspect metals. In addition to the usual cations and anions, determine specific gravity and conductivity. Check to see if the anion component of **MBD-15-SC** floats in the solution. Compare our results with the typical analysis the customer provided. If our analysis does not match theirs with reasonable precision, we will need to discuss this issue with them before setting up the columns. Final decisions regarding column sizes, expected throughput, and sampling intervals are to be reviewed and edited, if necessary, before proceeding.

COLUMN SET UP

Fill a 1-inch ID clear column with 160 ml's of **CG8-H** underneath 200 ml's of **WBMP**. Cut the top of the column down so that the screen inserts, when firmly inserted into the top of the column, almost touches the resin.

Fill a second 1-inch clear column with 350 ml's of **MBD-15-SC**. Cut the top of the column down so that the screen inserts, when firmly inserted into the top of the column, almost touches the resin.

Set up tube pumps to pump upward through each column at 25 mL/min flow. The expected specific gravity of the solution will cause the anion components to float, therefore an up-flow packed bed configuration is selected. Connect the effluent to a conductivity cell set up to data log.

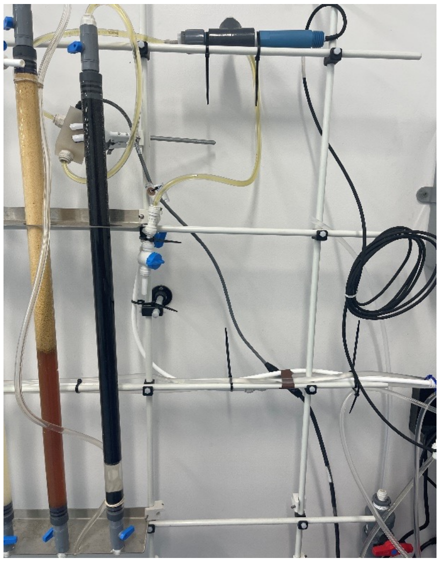
Rinse each column up flow with 3 liters of DI water, leaving the columns wet.

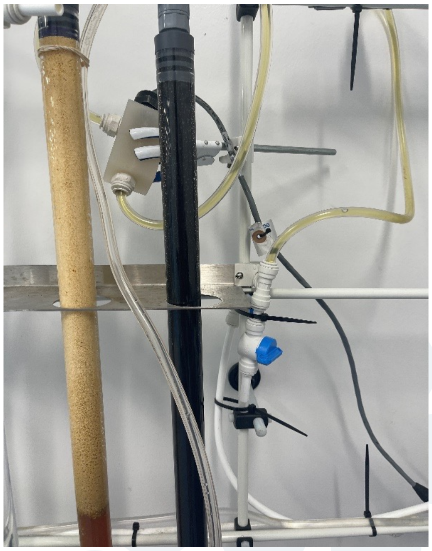
Switch to the client's solution and continue to run up flow until either conductivity substantially increases in the column effluent, or 10 liters of solution has been pumped through the column, whichever comes first. Take the first sample after 500 ml's, another at 1000 ml's throughput, and then sample every 1000 ml's. Test samples for phosphorous, manganese, copper, conductivity, and pH

DELIVERABLES

Charts for column exhaustion and leakage of targeted species. Preliminary process design. The information should be sufficient to allow the customer to begin the process of estimating capital cost and operating cost.

APPENDIX C: Photos





Find **APPENDIX D: Raw data** with the included Excel spreadsheet.