LIQUID PROCESSING
FILTRATION
ION EXCHANGE

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presented at
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RADIOACTIVE WASTE MANAGEMENT FOR
NUCLEAR POWER REACTORS
AND OTHER FACILITIES

University of Maryland at Baltimore
Baltimore, Maryland
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What's In Waste Water (Besides H_2O)

uspended Solids

1. Course particulate >1μ
   e.g. car rust, bic pens, sand
2. Colloids <1μ
   e.g. clay, silica, Co-58/60, Ag110m
3. Bacteria
   Grow great on ion exchange resin

B. Ionic

1. Dissociated Ions
   a. Cations
      e.g. Na^+, Co^{+2}, Cs^+, Ca^{+2}
   b. Anions
      e.g. Cl^-, SO_4^{-2}, I^-, $H_2BO_3^-$
2. Undissociated molecules
   e.g. H_3BO_3 BORIC ACID
3. Complexes
   e.g. EDTA(-4), EDTA-Co(-2)
   DECON WORK

C. Non Ionic

1. Dissolved
   a. Soaps
   b. Ethylene glycol, dry cleaning fluid
2. Non Dissolved
   a. Oil
   b. Grease

*SHORTENS FILTERS LIFE

* TECHNICALLY, BURIAL PROBLEM.

How to Remove the Bad Guys

1. Solids

   Filtration
   Precipitation
   Settling
   Coagulation

2. Ions

   Ion exchange
   Reverse Osmosis
   Precipitation

3. Oils, Soaps, Organics, Complexing Agents

   Stop at the Source
   Absorbtion
   Segregation of Streams

Filtration - The last Frontier

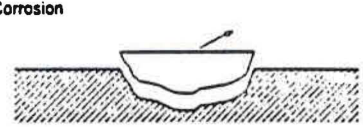
(10% of the activity = 90% of the headaches)

1. Typical Isotopes

Isotopes	% Particulate
Co - 58	0 - 95%
Co - 60	0 - 95
Cr - 51	50 - 95
Mn - 54	5 - 90
Nb - 95	30 - 90
Ag - 110m	90 - 100
Cs - 137/134	0 - 2
I - 131	0 - 2

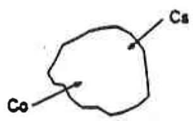
Sources of Particulates (1)

1. Corrosion



- high grade rust
- primarily Fe_2O_3 or some other iron oxide containing Co - 58/60, Cr - 51 & Mn - 54

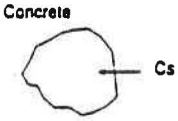
2. Resin



- Bead fracture or powdex
- any isotope
- not radioactive unless isotopes attach

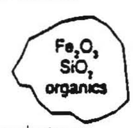
Sources Of Particulates (2)

3. Dust, Clay, Concrete



- any isotope
- not radioactive unless isotopes attach

4. Feed Water Colloids



- any isotope
- not radioactive unless isotopes attach

5. Bacteria

- Usually not radioactive

Note

- isotopes yield activity release
- all particles add to filter burden & clogging

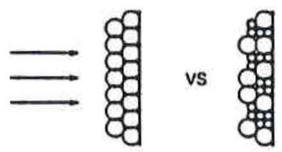
Particulate Properties (1)

1. Size

   - determine filter sizing
   - usual unit is the micron (μ)
     1(μ) = 0.00004 in
     = 0.0001 cm
   - see chart for sizing
   - filtration to 5μ is easy
     0.1μ difficult
     <0.1μ extremely difficult

2. Uniformity

   - the more uniform the higher the loading



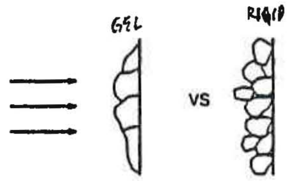
Particulate Properties (2)

3. Quantity

   - the more you have the quicker the filter plugs
   - measure as Total Suspended Solids (TSS)
   - activity not important to plugging
   - generally TSS is 0-10 ppm
   - can go > 10,000 ppm during crud burst, sludge treatment or resin sluicing

4. Rigidity

   - non rigid particles plug faster
   - backwash/cleaning easier for rigid particles
   - Fe_2O_3 and bacteria are not rigid



Particulate Properties (3)

5. Radioactivity

   - exposure
   - release
   - need not be related to plugging time
   - airborn problems on drying

6. Charge

   - agglomeration rate
   - removal efficiency
   - varies with filter

Note

   - Many of the above properties change with time. This can be used to improve processing. It also means real time testing on real streams IS a must.

Colloids

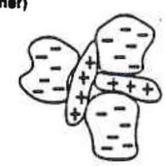
1. Small particles 1.0 - 0.01μ
2. Usually charged
3. Charges keep the particles from agglomerating

4. Charges attract other ions

5. Frequently composed of iron oxide (Fe_2O_3) and silica(S_iO_2)
6. Often from corrosion of stainless steel. These colloids in a reactor will contain Fe-59, Co-58, Co-60, Cr-51 and Mn-54
7. Ag 110m is usually 80-95% colloidal

How To Get Colloids

1. Filter them out.
   Problem: They are very fine and require ultra filtration or reverse osmosis.
2. Let them settle out.
   Problem: May take years.
3. Ignore them.
   Problem: They are a major source of radioactivity.
4. Coagulate them using a coagulant (allum, ferrous sulfate, polymer)



Problem: Too much of a good thing can disperse the colloids

Too little does not do the job

Each solution must be tested

Filtration Goals

1. Maximize removal
2. Minimize waste
3. Minimize cost
4. Minimize exposure
5. Maximize run time (filter life)
6. Minimize manpower

TERMINOLOGY

Filters

1. Cycle time - how long can it go without plugging
2. Sizing - what size particles will it remove
3. Sharpness of rejection - what percentage of a given size particle will it reject
4. ΔP (delta P) - pressure buildup across the filter - usual cause for removal from service
5. Backwash - reversal of flow direction to clean filter
6. Flow - how much water can be pushed through for a given ΔP

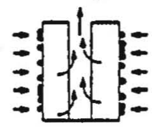
Filters

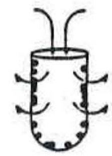
Murphy's Law of Filtration: If a filter will remove the particles that need removing, it will plug in 2 minutes or less.

Corollary: If it doesn't plug it won't remove anything either.

Filter Types

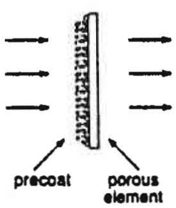
1. Cartridge - Disposable - 1-20μ





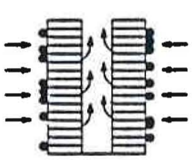
Filter Types (continued)

2. Precoat FINE PARTICULATES - BUT VOLUME INCREASE



precoat can be
- a. powdered resin
- b. diatomaceous earth
- c. any fine material

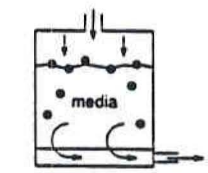
3. Etched Disk



a stack of etched disks can be used with precoat

Filter Types (continued)

4. Depth CAN TRAP ~ 0.1 μ



sand or carbon used
coarseness can be varied

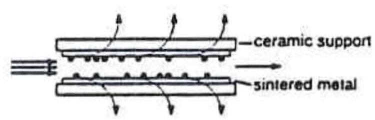
5. Membrane

- membrane openings can vary
  reverse osmosis 0.001μ>
  ultrafiltration 0.1μ - 0.001μ
- can be used for molecular sepation
- can be formed as sheets or hollow fibers

SHORT LIFETIME FOR HIGH LOADING

Filter Types (continued)

6. Cross Flow



SELF, CONTINUOUS FLUSHING

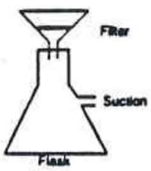
Filter Strengths (and Weaknesses)

Filter	Strength	Weakness
1. Cartridge	Disposable Variable size Good removal	Expensive Moderate waste volume Exposure on changeout >1μ only
2. Precoat	High flow Backwashable Good filtration to 1μ	High waste volume Ubiquitous precoat >1μ only
3. Etched Disk	High flow Low waste volume Backwashable	Hard to clean Low cycle times
4. Depth	Good removal Backwashable Can go to 0.1μ	Not absolute Moderate waste volume
5. Membrane	excellent removal removes colloids	Plugging/fouling Expensive Membranes subject to attack Reject waste stream
6. Cross Flow	excellent removal variable size	Reject waste stream Fouling

How To Know What's In Your Water

Particulates

1. Graded Filters



- Use several filters over desired range
  e.g. 10μ, 5μ, 2μ, 1μ, 0.45μ, 0.22μ
- inexpensive
- count filter papers (geometry)
- count influent and effluent
- use the effluent from one test as the influent for the next finer filter
- definitely worthwhile

2. Particle Sizing Instrumentation
   - expensive
   - distribution problem

   *ASSUMES DISTRIBUTION

Typical Wastewater Analysis (1)

1. Non-Radioactive Species

Species	ppm	meq / l
Na^{+}	60	2.61
Ca^{+2}	5.4	0.27
Mg^{+2}	6.7	0.56
Cl^{-}	82	2.31
SO_4^{-2}	20	0.42
B	240	22.2
SiO_2	5	0.08
TOC	4	

Conductivity 415μmho (μS)

pH 6.7

Typical Wastewater Analysis (2)

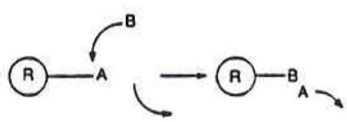
1. Radioactive Species

Species	μCi/cm³	meq/ l
Cr-51	1.4 E-4	9 E-11
Mn-54	1.8 E-5	8 E-11
Co-58	8.0 E-4	9 E-10
Co-60	2.5 E-4	1 E-8
I-131	1.8 E-4	1 E-11
Cs-134	1.0 E-3	6 E-9
Cs-137	1.5 E-3	1 E-7

Ion Exchange Basics

A. Reactions

1. General



2. Cation exchange

   A is usually H^{+}, Na^{+}, NH_4^{+} (Li^{+})

3. Anion

   A is usually OH^{-}, Cl^{-} ($H_2BO_3^{-}$)

B. Neutralization

1. (R)— H^{+} + Na^{+} ⟶ (R)— Na^{+} + H^{+}
2. (R)— OH^{-} + Cl^{-} ⟶ (R)— Cl^{-} + OH^{-}
3. H^{+} + OH^{-} ⟶ H_2O (DI water)

DE-IONIZED WATER

Ion Exchange Basics (continued)

C. Regeneration

Force the media into its' original form by using high concentrations of the proper ion.

e.g. H_2SO_4 can reverse reaction B.1.

NaOH can reverse reaction B.2.

D. Selectivity

The preference for one ion over another

e.g. clays prefer Cs+ over Na+

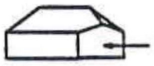
It arises from:

- charge to size ratio (e.g. organics)
- size (e.g. zeolites)
- chemistry (e.g. chelating resins)
- solubility (e.g. Cl- on Ag)

Types of Materials

1. Organics - bead, macroreticular

2. Zeolites - crystalline clays, artificial



3. Glass based - high surface area

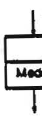
4. Carbon based - high surface area
5. Inorganic oxide - high surface area

Properties of Exchangers

1. Size (bead vs powder)
2. Selectivity
3. Temperature Resistance (structural, functional)
4. Capacity
5. Mechanical strength
6. Purity (standard vs nuclear grade)

Ion Exchanger Performance

Capacity - amount of ion that can be removed



- usually quoted in equivalents/liter or meq/ml
- typically in the range
  2 meq/ml for organic cation
  1.3 meq/ml for organic anion
  * NOT FOR SELECTIVE MEDIA
- not very meaningful for selective media

Ion Exchanger Performanance

Calculations Involving Capacity

a. capacity for Na^+

eq wt = at wt/charge = 23/1 = 23g

cation resin capacity = (2eq/l)(23g/eq) = 46 g/l

b. capacity for Ca^+

eq wt = at wt/charge = 40/2 = 20g

cation resin capacity = (2eq/l)(20g/eq) = 40 g/l

Ion Exchanger Performanance

Throughput

- volume of water that can be treated by a given volume of media
- typically in gal/ft^3
- often measured in column or bed volumes

gal/ft^3 = 7.5 · bed volumes

Ion Exchanger Performanance

Throughput Calculations

What is the throughput expected for wastewater that contains 40 ppm Na. Assume the capacity of the cation exchanger is 1.9 meq/ml (eq/l).

The water contains 40 ppm Na = 40mg Na/l water

$$40\text{mg Na/l water} = \frac{40\text{mg/l}}{23\text{mg/meq}} = 1.7 \text{ meq Na/l water}$$

the throughput for the cation resin is :

$$\frac{1.9 \text{ eq/l resin}}{1.7 \text{ meq/l water}} = \frac{1.9 \text{ eq/l resin}}{1.7 \times 10^{-3}\text{eq/l water}}$$

= 1100 l water/l resin

= 1100 column volumes

= (1100)(7.5) gal/ft³

= 8250 gal/ft³

Ion Exchanger Performanance

Some Notes On Throughput Calculations

- if more than one cation exists you must sum the meq/l of all the cations and use the sum to calculate the capacity
- in any solution the total meq/l of cation must equal the meq/l of anion * MIXED BEDS
- if the solution is neutral pH then ↑

$$\frac{\text{throughput cation}}{\text{throughput anion}} = \frac{\text{capacity cation}}{\text{capacity anion}}$$

- calculations for selective media are next to impossible
  - CAN ONLY TEST RUNS

Ion Exchanger Performanance

Decontamination Factor (DF)

$$DF = \frac{\text{influent concentration}}{\text{effluent concentration}}$$

e.g.

Cs = 3.0×10^{-3} µCi/ml	influent
1.0×10^{-4} µCi/ml	effluent

$$DF = \frac{3.0 \times 10^{-3}}{1.0 \times 10^{-4}} = 30$$

This says 1/30 of the Cs is getting through.

- LARGER DF, THE BETTER

Ion Exchanger Performanance

Residence Time

$$\text{residence time} = \frac{\text{volume of bed}}{\text{rate of flow}}$$

- length of time for exchange
- the longer the better
- watch your units during calculation

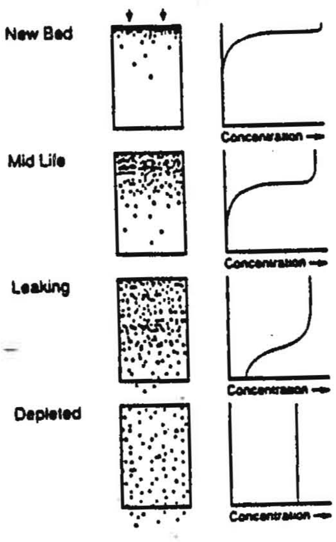
e.g.

for a 50 cu/ft bed at 30 gpm

$$\text{residence time} = \frac{(50 \text{ ft}^3)(7.5 \text{ gal/ft}^3)}{30 \text{ gal/min}}$$

=12.5 min

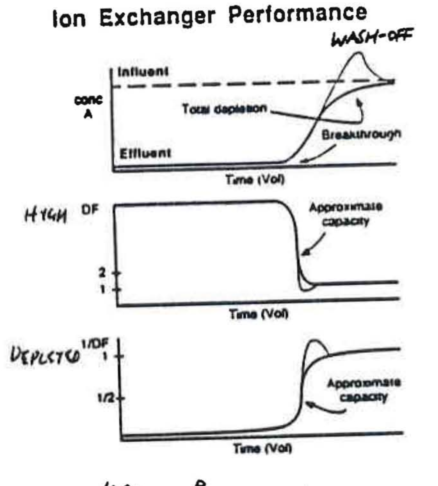
Ion Exchanger Performance



* SET TOLERANCE LEVEL

1/1000, etc.

Ion Exchanger Performance



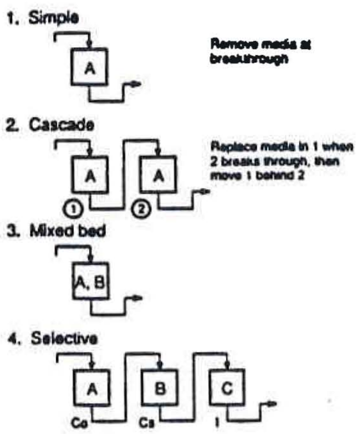
* SLOW FLOW WILL ACCOUNT FOR RESIN RESPONSE BETTER.

TRACKING OF ION EXCHANGERS

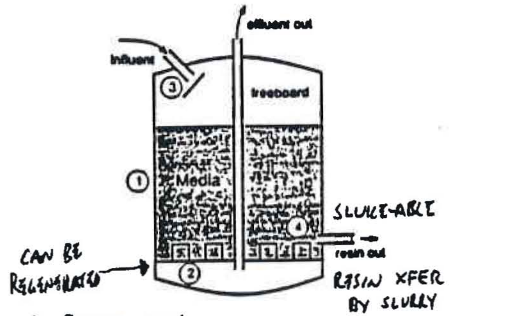
pH & CONDUCTIVITY : BY WATCHING pH IN CATION BEDS
- DEPLETED WHEN BOTTOM REACH NEUTRAL (?)

↓ MIXED BEDS
EFFLUENT CONDUCTIVITY SAME AS INFLUENT.

Bed Use



Hardware



1. Pressure vessel
2. Retention scheme (tube sheet or hub and lateral)
3. Influent distributor (screen, plate or hub and lateral)
4. Resin out
(5.) Regeneration equipment

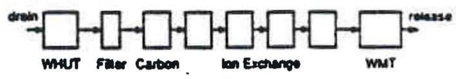
Typical dimensions:

Typical dimensions:	Height	5-7 feet
	Diameter	2-4 feet
	Volume	20-60 cu ft

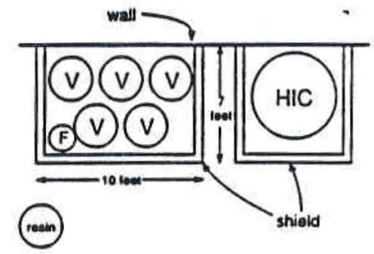
*PRESSURIZED SYSTEMS ARE MORE EFFICIENT

Filtration - Ion Exchange System

1. Typical Schematic



2. Typical Layout



Ion Exchange - Filtration

1. What's in the water — ALWAYS ANALYSE FIRST
   Solids - size, nature, amount
   Ions - what kinds, how many
   Non ionics - oils, etc.

   Use filter tests, conductivity, pH, γ spectroscopy, atomic absorbtion (AA), inductivity coupled plasma (ICP), ion chromatography, total organic carbon (TOC), oil and grease
2. What must be removed
   Where is the water going
   Selective vs non selective
   DF needed
3. Flow rate
   Peak / normal
4. Vessels / Filters
   Size, number, in plant, mobil, sluicable
5. Will it work — ALWAYS TEST THE REAL WATER

Trouble Shooting Your Ion Exchange / Filtration System

	Problem	Possible Cause(s)
1.	Poor Co/Cs/Ag removal	a. Colloids b. Oil c. Complexes (decon fluid)
2.	Short bed Life	a. Oil b. Organics (soaps/detergents) c. High conductivity
3.	Short filter life	a. Too much sludge (Time to clean up the tanks) b. Wrong type of filter c. Crud burst
4.	Constant Co leakage	a. Colloids b. Soaps/detergents
5.	High Co levels in the WMT	a. Complexes (decon fluids)
6.	Constant leakage of all isotopes	a. Poor sluicing b. Poor regeneration

KNOW WHAT IS GOING ON IN THE PLANT

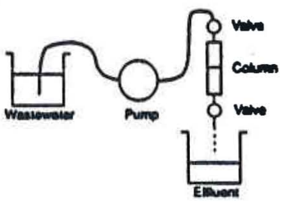
Start a Test Program

You need:

1. Knowledge
   - Other courses
   - Read books / literature
   - Attend meetings - EPRI/WM
   - Talk to vendors (technical section)
   - Talk to other utilities
2. Ion Exchange Set Up
   - Burets
   - Pumps
   - Valves
   - Buy one / make one
3. Filter Set Up
   - Graded filters
   - Vacuum pump
4. Friends
   - In the count room
   - In the chemistry lab
5. Evangelize
   - There is money to be saved
   - There is volume to be reduced
   - There is offsite dose to be reduced

Ion Exchange Test System (1)

1. Equipment



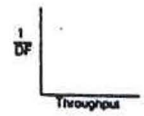
- need timer
- analysis required at regular intervals for effluent
- analyze influent
- scale flow to match residence time in real system
- column diameter >10 media diameters
- test real water, real time

Ion Exchange Test System (2)

2. Calculations

DF
Total Volume

OR



3. Results

- DF will probably be better in full system
- capacity to 1-2% of full system
- useful for media selection

Ion Exchange - Where to Learn More

1. Books

   Kunin "Ion Exchange Resins"
   Krieger Publishing Company

2. Courses
   Am. Inst. Chem. Engr.

3. Vendors

   a. Organic Resins
      Dow, Rohm & Haas, Mobay, Sybron/Ionac, Purolite

   b. Selective
      Mobay, Union Carbide, Steelhead, Duratek
      Zeolites Nat'l Zeolites

4. Me

Dr. Herbert G. Sutter
Scientific Applications
International Corporation
12850 Middlebrook Road
Suite 300
Germantown, Maryland 20874
(301) 601-1231

Useful Information

Measurement

1000 microns (μ) = 1 millimeter (mm) = 0.039 inch

100 μ is the width of a pen stroke

1 m³ = 35.3 ft³ = 1000 l

1 ft3 = 28.3 l = 7.48 gal

1 gal = 3.79 l

20 mesh	840 μ
42 mesh	350 μ
60 mesh	250 μ
100 mesh	149 μ

Ion	Atomic Weight	Equivalent Weight	Power Plant Source
Na^{+}	23	23g	salt, soap
Ca^{+2}	40	20g	concrete
Cl^{-}	35.5	35.5	salt
SO_4^{-2}	96	48	concrete