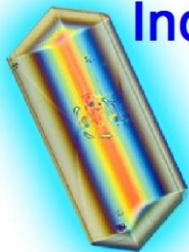


State of the Art of Eutectic Freeze Crystallization

Dr. Marcos Rodriguez Pascual



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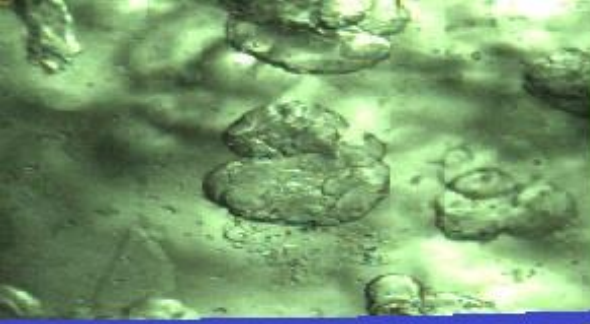
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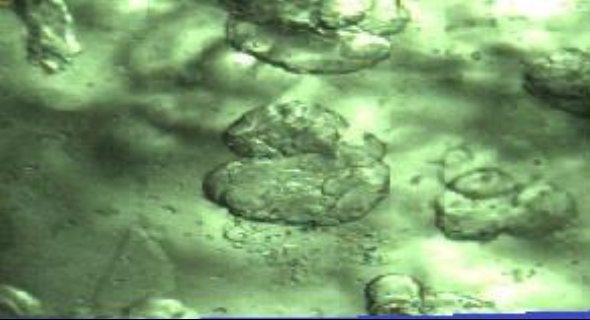
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Eutectic Freeze Crystallization

- IPS&EFC SEPARATIONS HAS DEVELOPED A CHEMICALS FREE, LOW COST, LOW ENERGY CONSUMPTION (EUTECTIC) FREEZE CRYSTALLIZATION PROCESS THAT CAN BE APPLIED TO REDUCE WASTE WATER VOLUMES, CONCENTRATE SALT SOLUTIONS, SEPARATE SALTS AND WATER, REDUCE WASTE WATER PROBLEMS AND CREATE ZERO LIQUID DISCHARGE OPERATIONS.
- APPLICATIONS FOR THIS ARE ACROSS A WIDE RANGE OF INDUSTRIES, SUCH AS MINING, OIL/GAS PRODUCTION, CHEMICALS PROCESSING AND THE WASTE WATER AND DRINKING WATER INDUSTRIES.
- IPS&EFC HAS DEMONSTRATION UNITS RUNNING AT 100-500 KG/H SCALE FOR THE FREEZE CONCENTRATION AND EUTECTIC FREEZE CONCENTRATION APPLICATIONS

Technology Development



**Bench
Scale
(10-15 L)**



**Pilot
Scale
(100 L)**

**Skid mounted
unit
(250 L)**

Industrial Scale

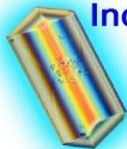


**Laboratory
Scale
(0.25-2 L)**

1995

2008

2012



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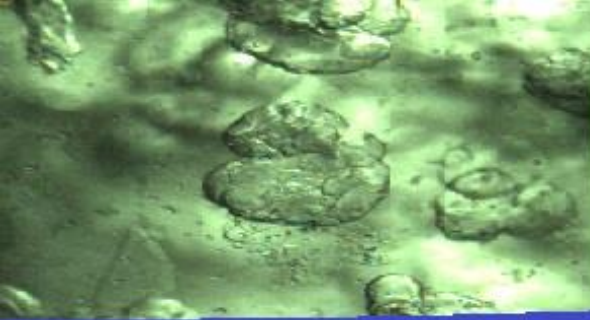
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High Salinity Water Treatment

Growth of fresh water
production increases high
salinity waste water streams

Drive for Zero Liquid
Discharge processing

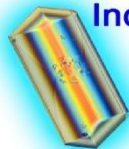
Disposal options for high
salinity water streams
decreasing and becoming
more limited and more
expensive

High salinity water treatment required
Market size >> 10,000 units globally

Recovery of water for re-use
from industrial waste streams

→ ZLD operations

Recovery of valuable salts
from waste water



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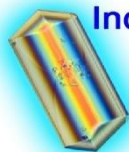
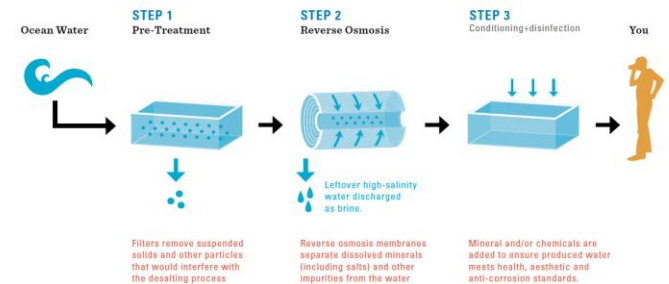
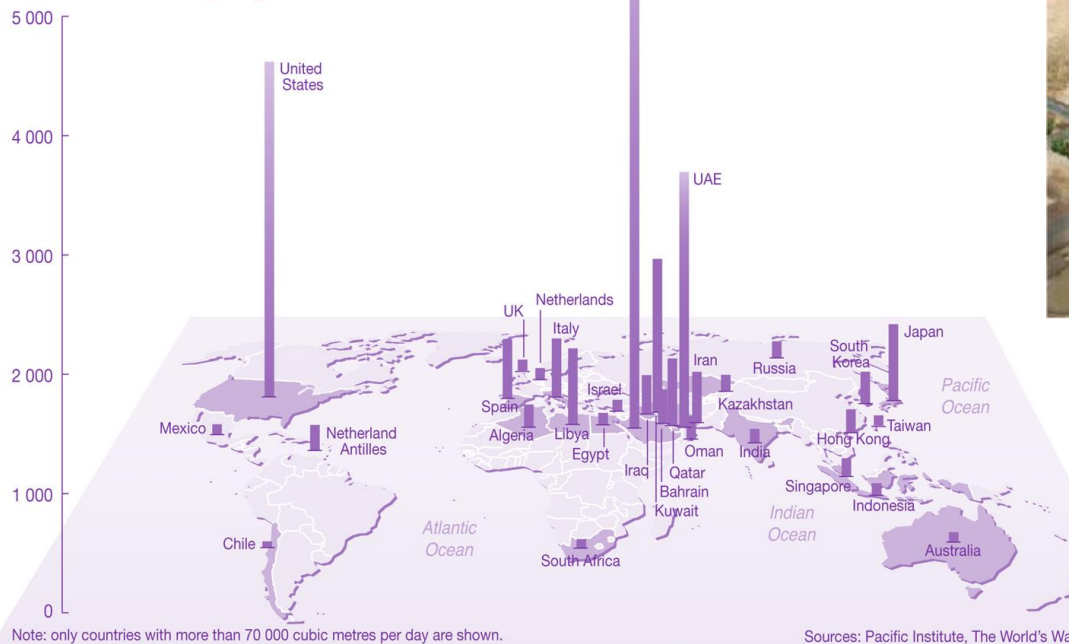
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Water scarcity and pollution

Desalination

Water desalination

Desalination capacity
Thousand of cubic metres per day



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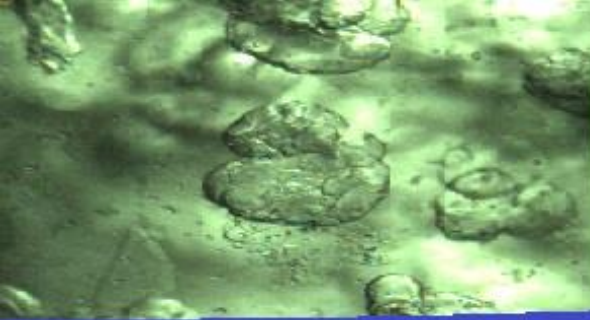
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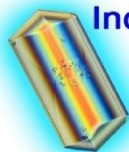
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Water scarcity and pollution

Industrial Waste Waters



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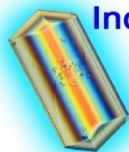
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Water scarcity and pollution

Mining



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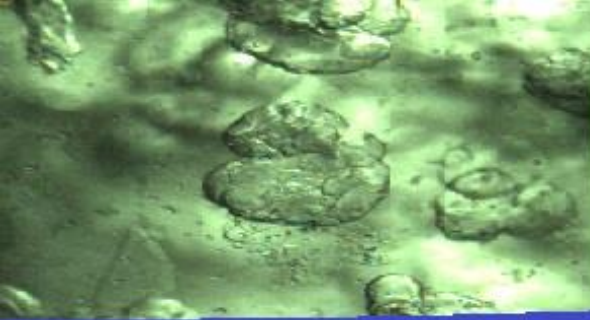
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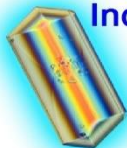
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Water scarcity and pollution

Acid Mine Drainage



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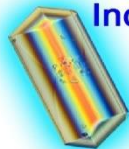
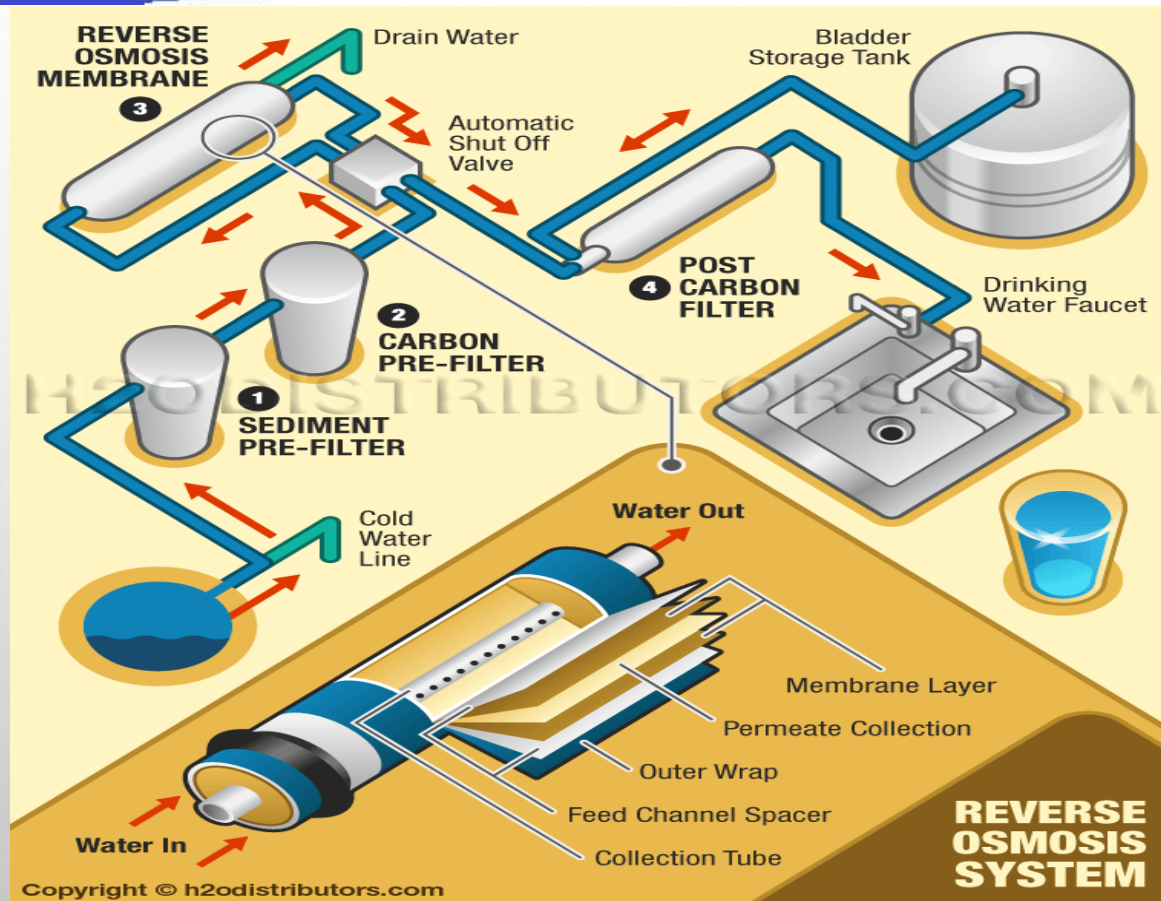
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What is the solution to this?



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Reverse Osmosis for Desalination

Sea Water/
Waste Water

Reverse Osmosis



http://img.natlacpa.com/image_nu/photo-g/reverse-osmosis-water-maker-for-ships-30946-3250113.jpg

H₂O

Brine



http://www.waters.com/webassets/cms/promotion/media/home_page_background/bg_image_water_woman.jpg

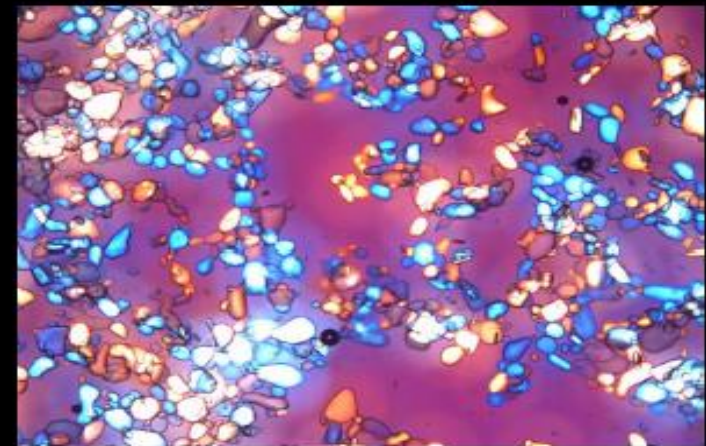
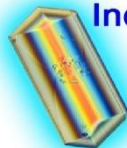


Photo: Tim Egan



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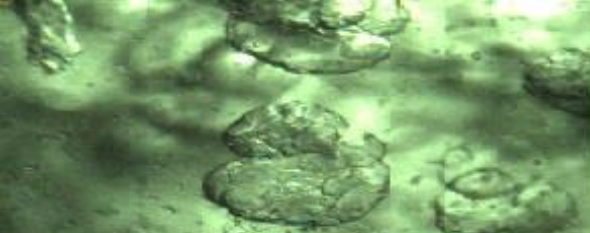
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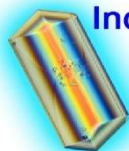
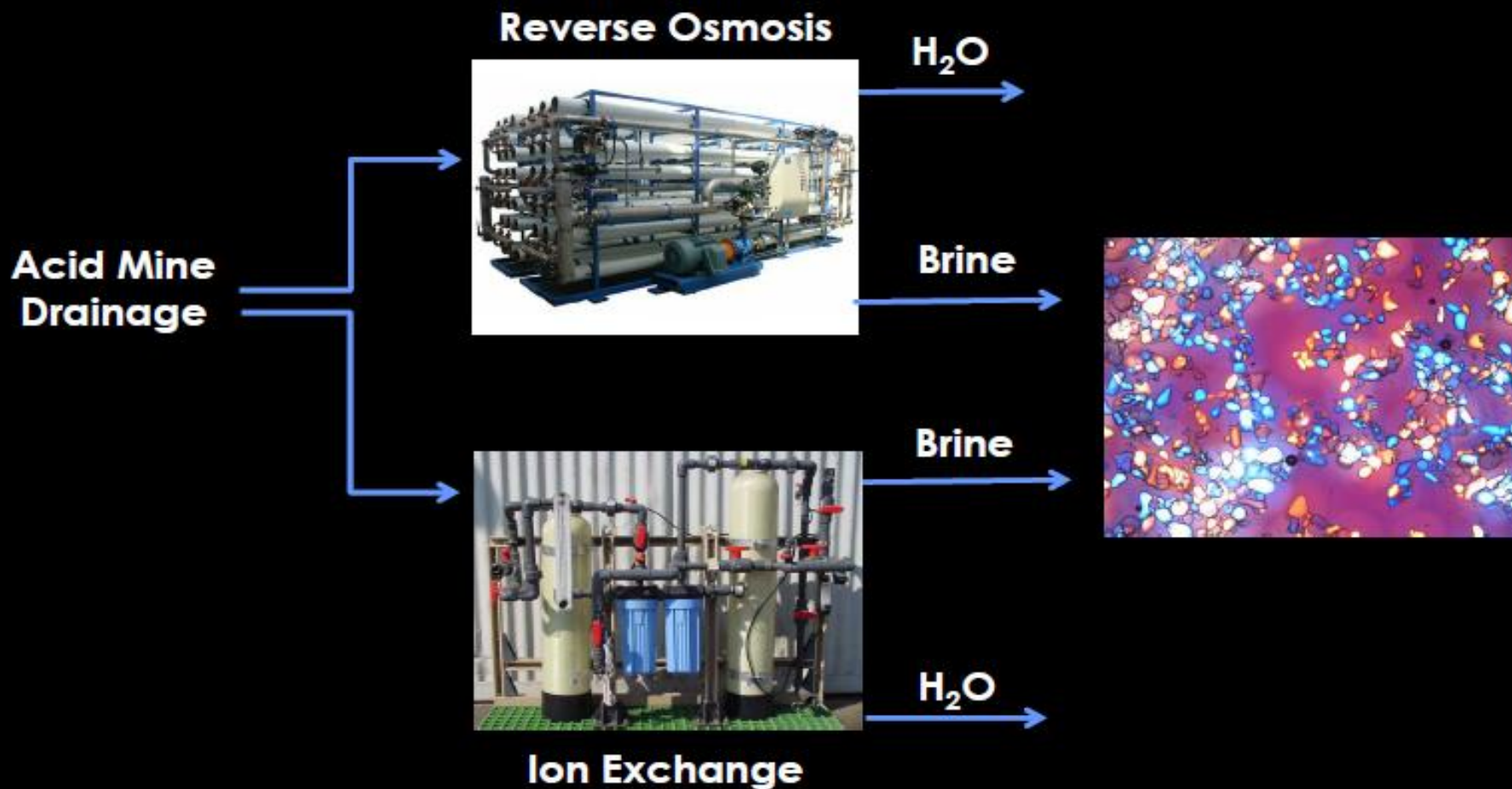
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RO + Ion Exchange for AMD



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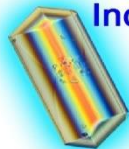
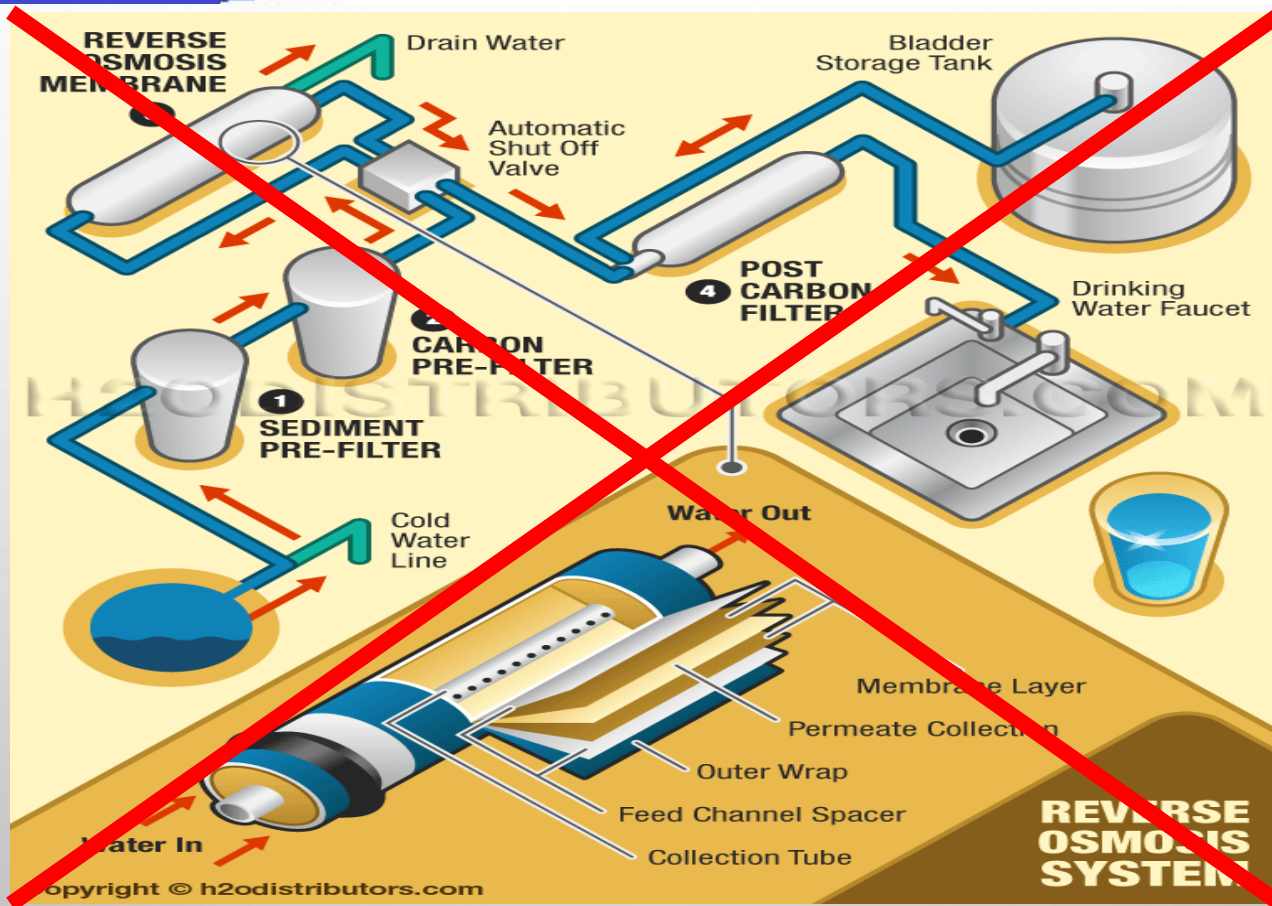
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And The concentrated Brine?



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South Africa's Brine disposal



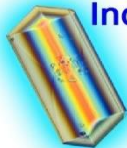
**Lined evaporation
ponds**



**Mechanical
evaporators**



**Disposal to ash
systems**



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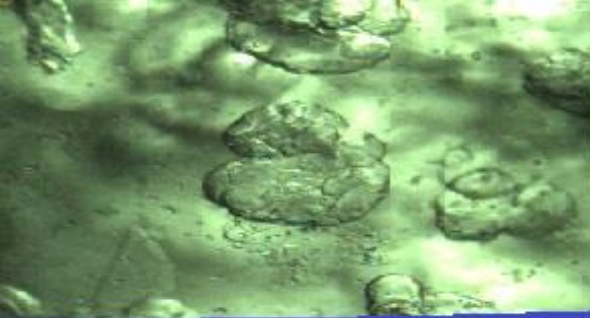
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High Salinity water streams are created in a wide range of industries

Mining and minerals

- Acid mining drainage
- Salt recovery
- Water recycle
- Waste water

Oil & Gas production

- Co-produced water
- Fracking and tar sands processing
- Waste waters
- Gas scrubbers

Chemical Industry

- Neutralization fluids
- Scrubbers
- Dye industry
- Salt production
- Recovery of salts
- Recovery of organic materials

AgriCulture

- Manure processing
- Digestate biogas
- Recovery of organic molecules

Waste water industry

- Reduce salt load to bio-treatment
- high salinity streams

Dairy Industry

- Cheese brine dewatering

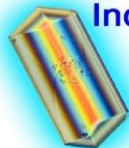
Metals processing

- Pickling liquids
- Acid Recycle

Drinking Water

- RO concentrate
- IEX regenerate
- Thermal evaporation brines

These applications have urgent, regulatory AND cost driven issues



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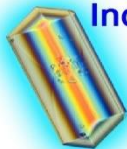
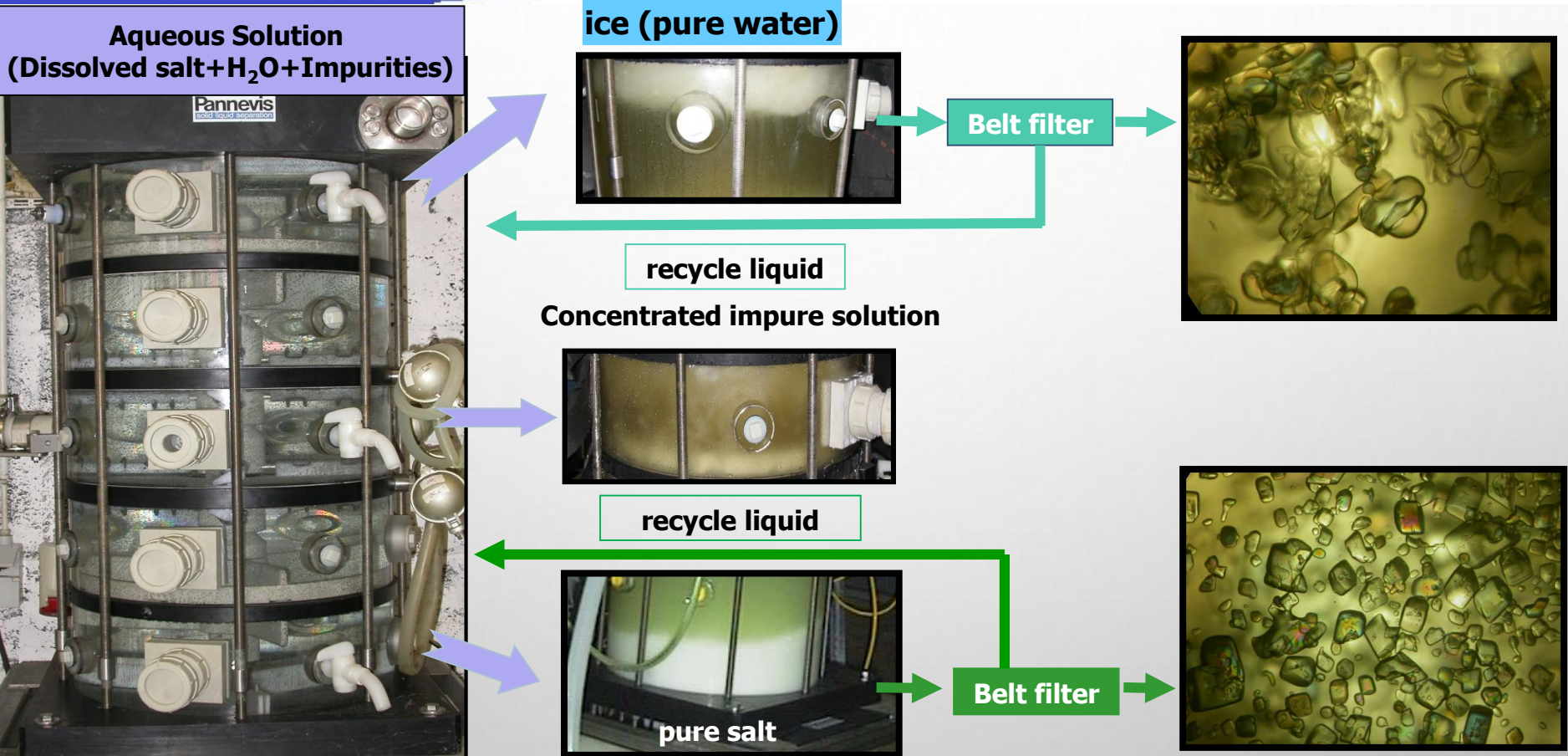
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EFC Technology Principle



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No additional chemicals

NEED MORE CHEMICALS?



The chemicals, equipment and apparatus in your Gilbert Chemistry Set are complete in every respect to perform all of the experiments for which the set is intended. However, as you use the set, your chemical supplies will naturally become depleted and some of the equipment may need replacement.

When this happens, you may order additional supplies direct from The A. C. Gilbert Company, New Haven 6, Conn. Mark your envelope: "Attention Consumer Division." A price list is located elsewhere in this manual.

By ordering from the Gilbert Company, you will be receiving standard, official and safe chemicals exactly like those in your set originally.

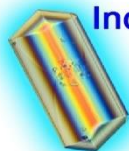
**DON'T RUN SHORT...
ORDER YOUR CHEMICALS
BEFORE YOU USE THEM UP!**

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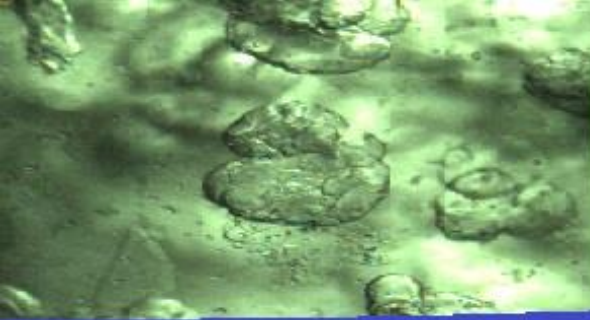
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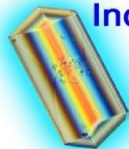
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Low temperatures results in less corrosion and the process is safe and easily controlled



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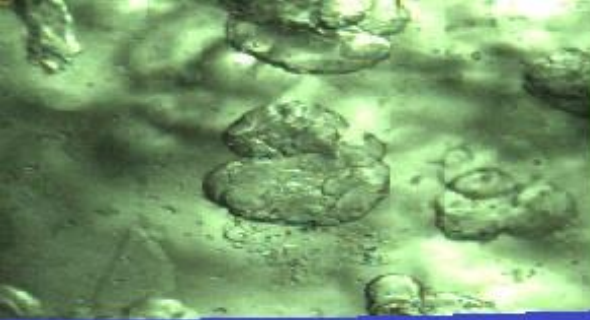
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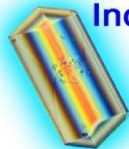
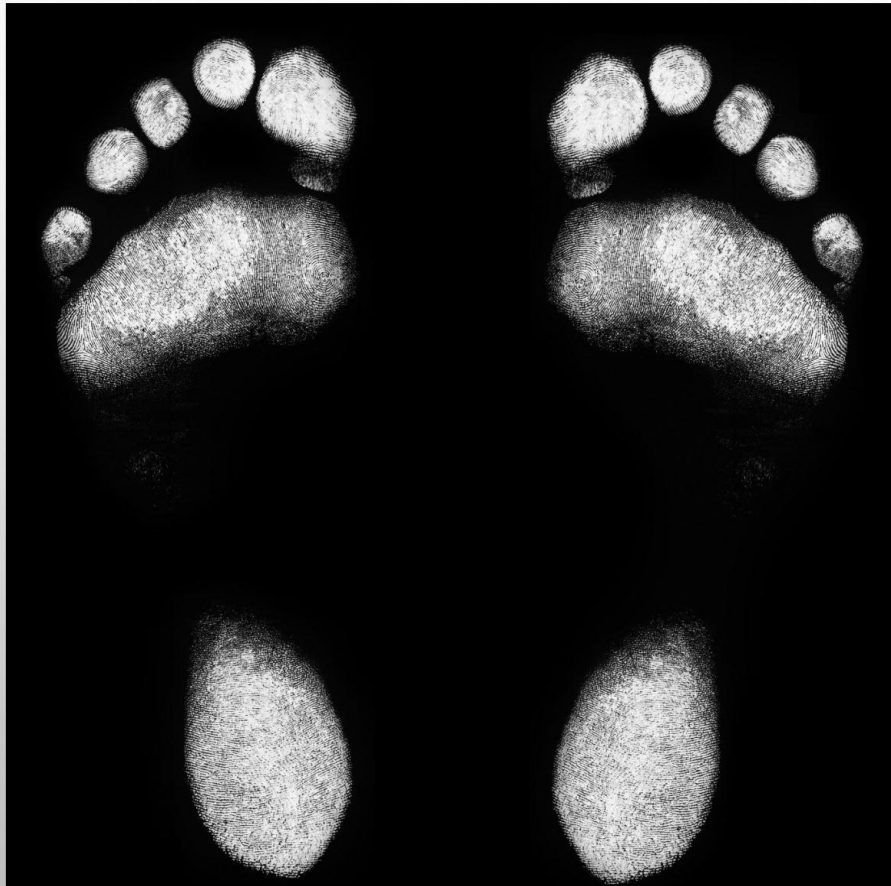
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Minimal Foot Print



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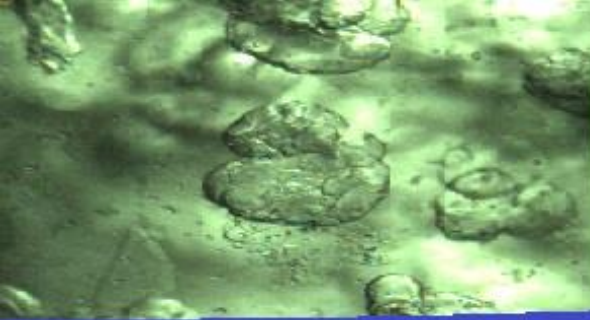
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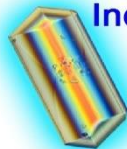
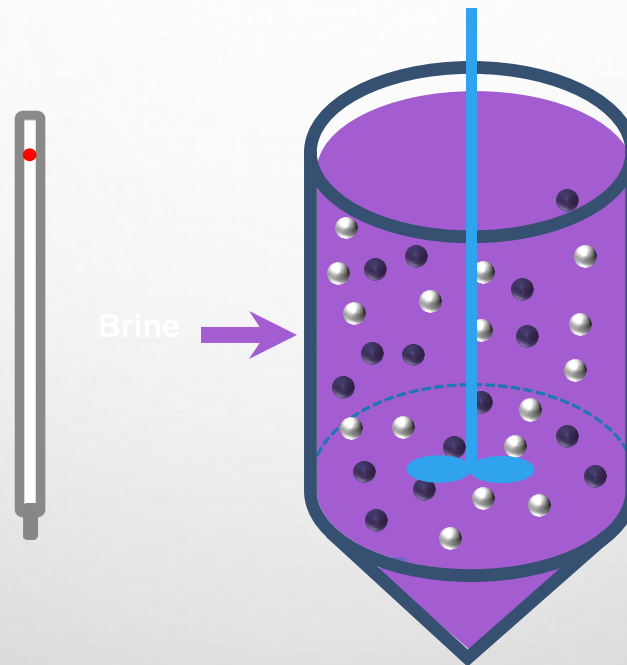
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Gravitational Separation



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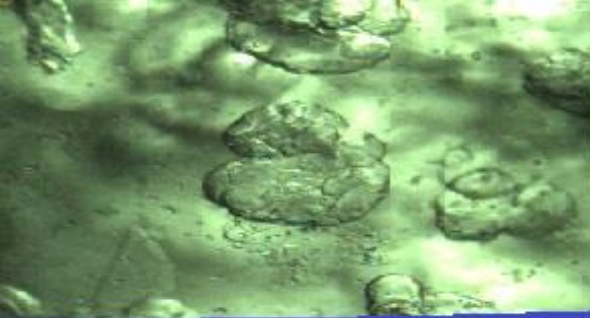
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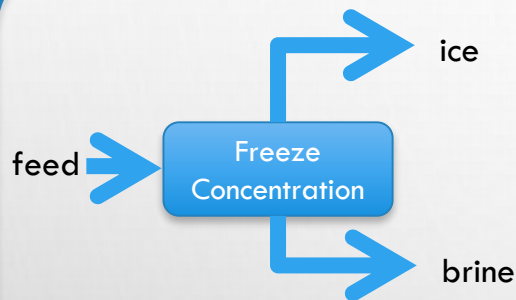
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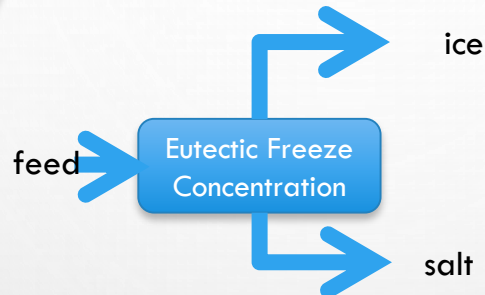
IPS&EFC SEPARATIONS OFFERS FREEZE CONCENTRATION (FC) AND EUTECTIC FREEZE CONCENTRATION (EFC) DEPENDING ON PROBLEM OF CUSTOMER



Freeze Concentration

- Reduces volume stream
- Produces clean ice and high salinity brine
- Removal of 50-80% water as ice
- Simpler type of crystallizer compared to EFC process

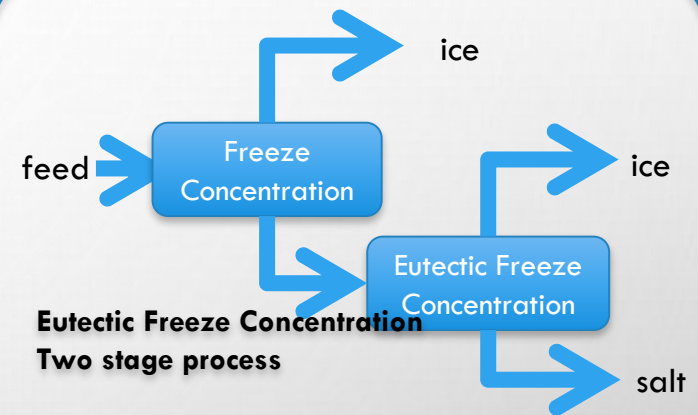
Use simple ice machine



Eutectic Freeze Concentration Single stage process

- Produces clean ice and clean salt
- Apply for feed streams closer to the Eutectic composition.

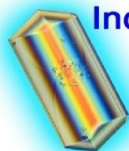
EFC crystallizer



Eutectic Freeze Concentration Two stage process

- Produces clean ice and clean salt
- Pre-concentration via freeze concentration to reduce EFC crystallizer size
- A similar process can be applied with cooling crystallization as pretreatment

Two stage process



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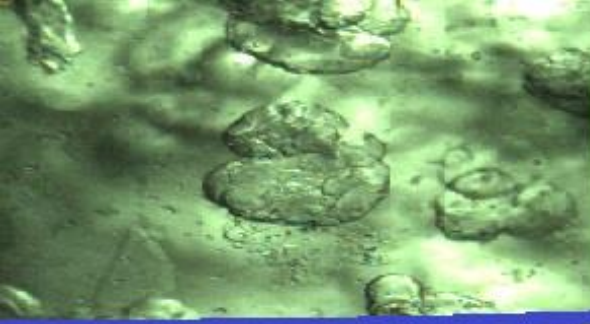
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EFC and FC have different Customers Drivers

Recovery

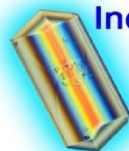
- Recovery of salts with high value or high disposal cost
- EFC as separation technology (salt, organic molecules)

Compliance

- Volume reduction of expensive waste water stream
- Removal of salts for disposal (easier as solid)

Water recycle

- Removal of water from waste stream for re-use



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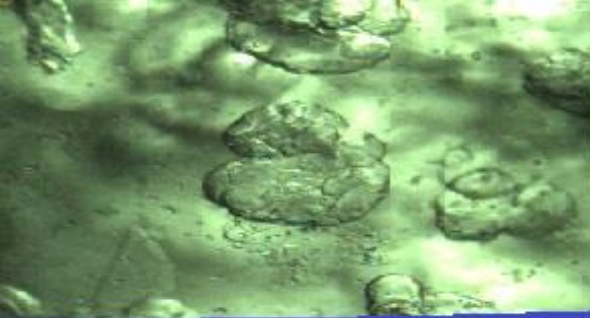
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Removing Salt from water is not easy

EVAPORATIVE TECHNOLOGY

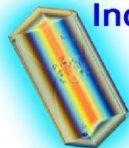
- Energy intensive
- New heaters can be permit restricted
- Corrosion and scaling tendency
 - High chemicals usage
- Difficult to operate
- Cannot deal with heat sensitive products
- Limit on inlet salt concentration

MEMBRANE FILTRATION

- No salt recovery, always a brine
- Scaling tendency high
- Limit on inlet salt concentration
- Problematic for high COD waters

EFC

- Low energy cost
- No chemicals for scaling & corrosion
- Can handle corrosive liquids
- Easy to operate
- No inlet limit
- Scaling not an issue
- Can handle heat sensitive products



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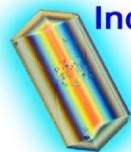
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EFC SEPARATIONS HAS A STRONG CUSTOMER BASE FOR PAID APPLICATIONS AND PILOT PLANT RESEARCH

A selection of Recurring customers → these customers operate evaporators and membrane systems but they come to IPS&EFC Separations with the problematic streams



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EFC is READY for Commercial Scale

- TECHNOLOGY PROVEN AT 1L, 10L, 100 L/H, 500 L/H SCALE WITH >100 DIFFERENT APPLICATIONS
- DEMONSTRATION UNITS RUNNING IN SUMMER 2015 WITH VALIDATED OPERATING AND MANUFACTURING COST

EVIDES ROTTERDAM
KUUNDERS, DEURNE
CONF CUST, NL

100 KG/H EFC UNIT ON ION EXCHANGE REGENERATE RECOVERY (ZLD)
500 KG/H FC UNIT ON RO CONCENTRATE FROM MANURE INDUSTRY
300-500 KG/H FC UNIT FOR CONCENTRATION OF FOOD PRODUCT STREAM

MAIN EQUIPMENT AVAILABLE AND WELL PROVEN IN SMALL AND LARGE CAPACITY:

ICE AND COOLING MACHINES

BELT FILTERS

CRITICAL EQUIPMENT: CRYSTALLIZER DESIGNS EFC (PATENT) → VERY SCALABLE, MODULAR SYSTEM, PROVEN MANUFACTURING

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EFC HAS A SOLID BUSINESS CASE

Example: waste water stream with various salts (corrosive and scaling)
volume reduction prior to disposal

	Evaporator	FC	Disposal	Column2
Capex	550	590		kEuro
Opex (incl depreciation)	246	144		kEuro
Total Cost of Ownership	24	14		Euro/m3
			35	Euro/m3
Pay Back time	4.6	3.2		year

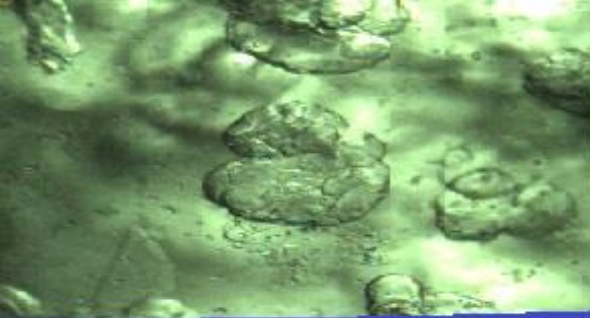
Attractive economics
for service model

Higher operating cost
evaporator due to energy
costs (50% higher),
downtime, corrosion, scaling

3 year payback
acceptable for
environmental
investments

Disposal cost at waste
water facility
Uncertainty how long this
can be allowed

Costs taken from commercial quotes



FIELDS OF APPLICATION

As a Separation Technology

Separation technology for the isolation of valuable products from an aqueous process.

Some examples of tested systems:

- Recovery of organic acids from fermentation broths.
- Isolation of various high quality salts for different producers has been studied (e.g. NaCl , MgSO_4 , NiSO_4)
- Salts from scrubbers
- Ion Exchange Regenerate for ZLD
- Producing valuable liquid concentrate products by freeze concentration

As a Waste water treatment

Removing components from a waste water stream to be reused or to facilitate Zero Liquid Discharge.

Some examples of tested systems:

- Treatment of oil and gas co-produced and flowback waters.
- Recovery of NiSO_4 from a metal recycling plant.
- Crystallization of soda from an incinerator blowdown stream.
- Volume reduction of liquid pig manure RO- concentrates by freeze concentration.
- Many different salts from various waste streams have been tested so far (e.g. Na_2SO_4 , $(\text{NH}_4)_2\text{SO}_4$, KBr)

EFC is not always an 'end-of-pipe' solution. Often an integration with other unit operations is required to come to the most optimal configuration. IPS&EFCseparations BV is actively involved in this optimal process configuration design work.

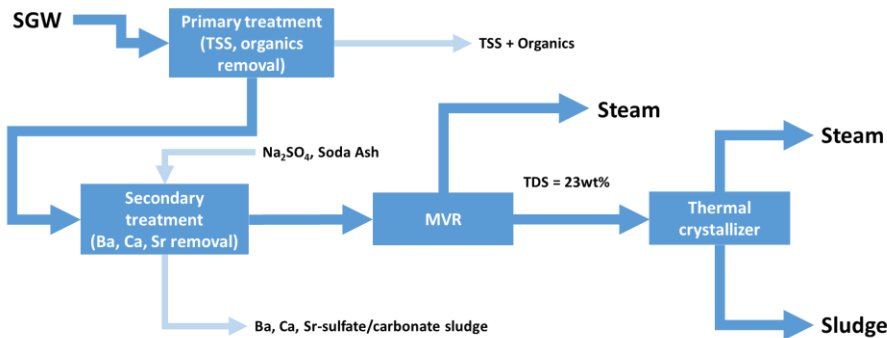
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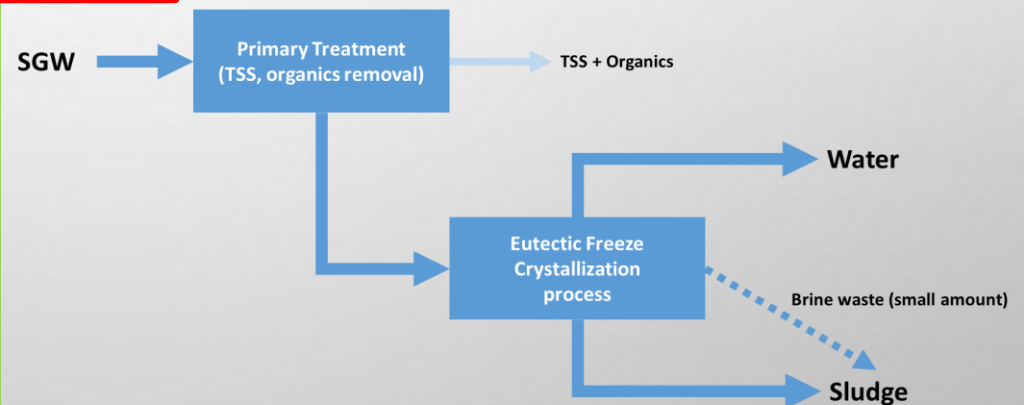
SHALE GAS WATERS → LOWER CAPEX/OPEX

Projects for a major oil and gas company and a North American waste water company

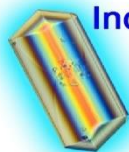
Scheme 1: THERMAL CRYSTALLIZER ROUTE



Scheme 2: EFC ROUTE



- Less process steps
- No additives for secondary treatment
- Less corrosion and safe process due to low operating temperatures
- Less energy consumption
- Flexible, easy capacity increase by modular EFC process design.
- >25% lower total cost of ownership



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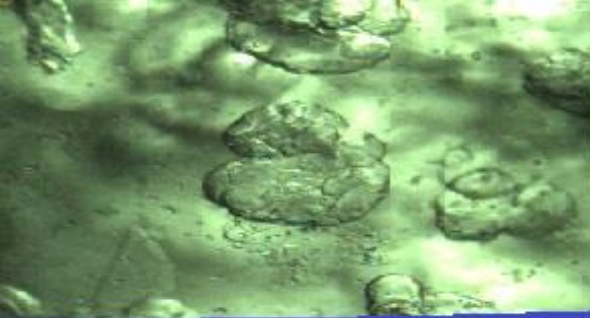
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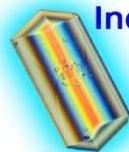
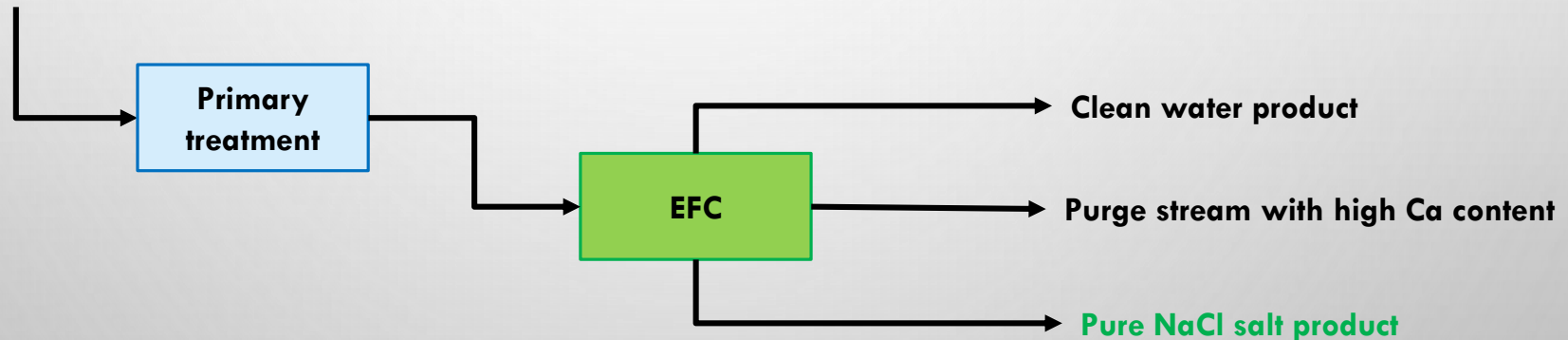
OIL CO-PRODUCED WATER

Project for an oil producer from the Middle East

- Removal of NaCl greatly reduces the negative effects of reinjecting or discharge of the co-produced waters.
- Less brine left for further treatment (in evaporation pond) or reinjection.
- EFC process produces clean water from the oil waters that can be used on site. Big advantage in water scarce areas.



Oil co-produced water



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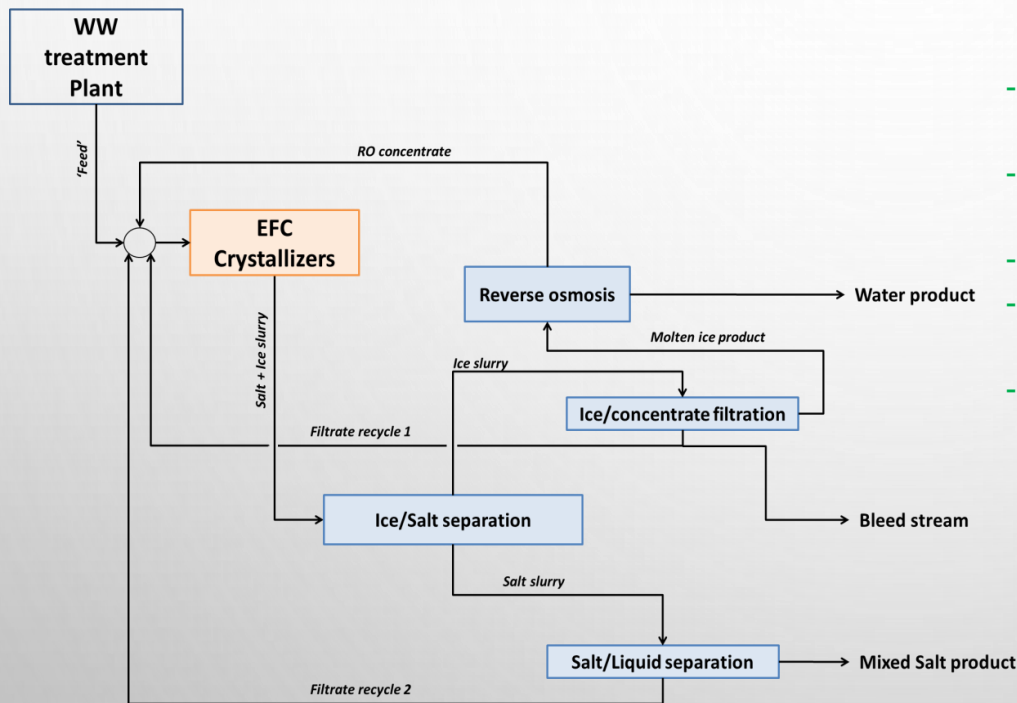
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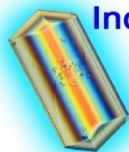
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ACID MINE DRAINAGE TREATMENT

In collaboration with South African engineering firm and University of Cape Town



- Large liquid waste volume reductions of current AMD treatment facilities can be achieved.
- Mobile containerized units can be build to treat existing AMD reservoirs.
- Applicable to wide range of AMD compositions
- Demand for proper AMD treatment systems is rapidly growing.
- Stricter legislation due to the severe environmental risks associated with current situation.



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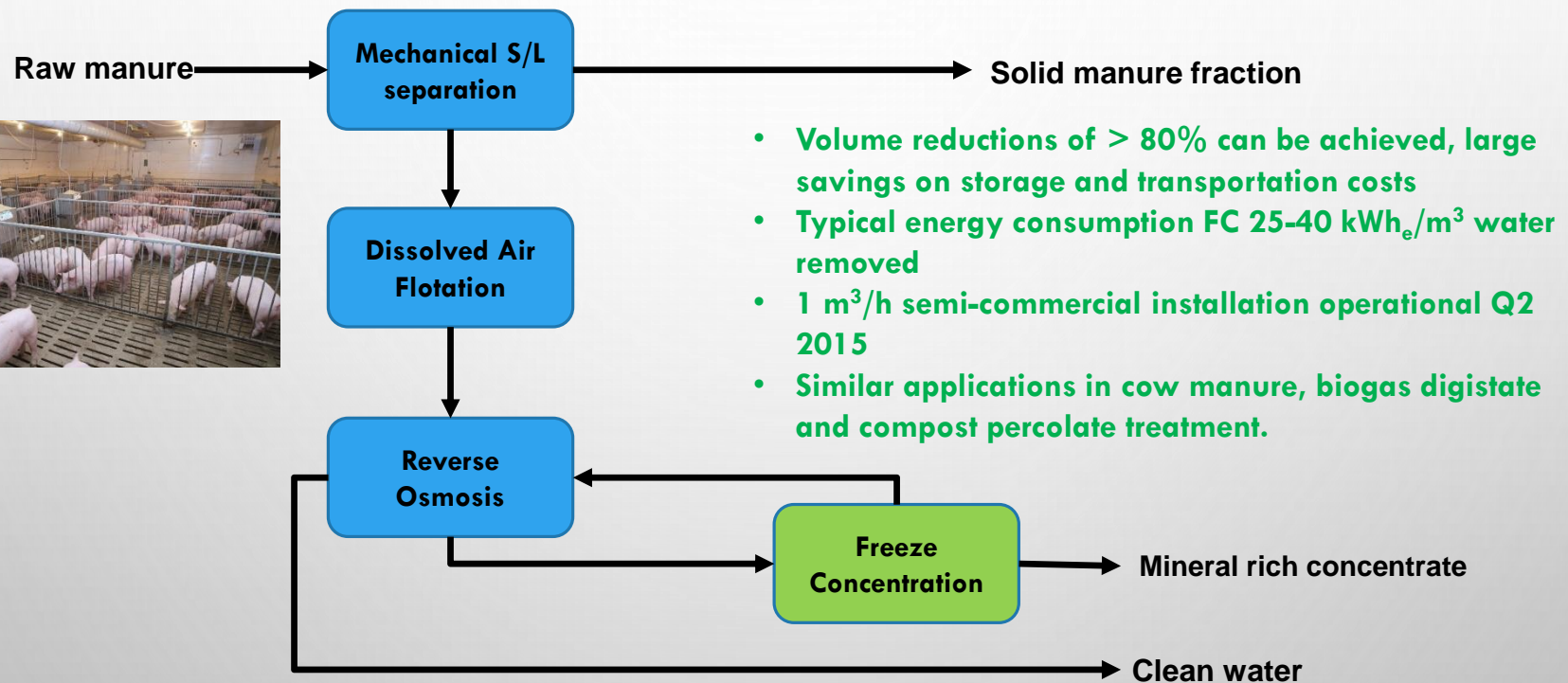
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LIQUID MANURE

Projects with pig farmers, manure treatment company and specialized manure installation company

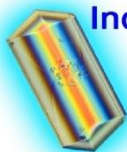
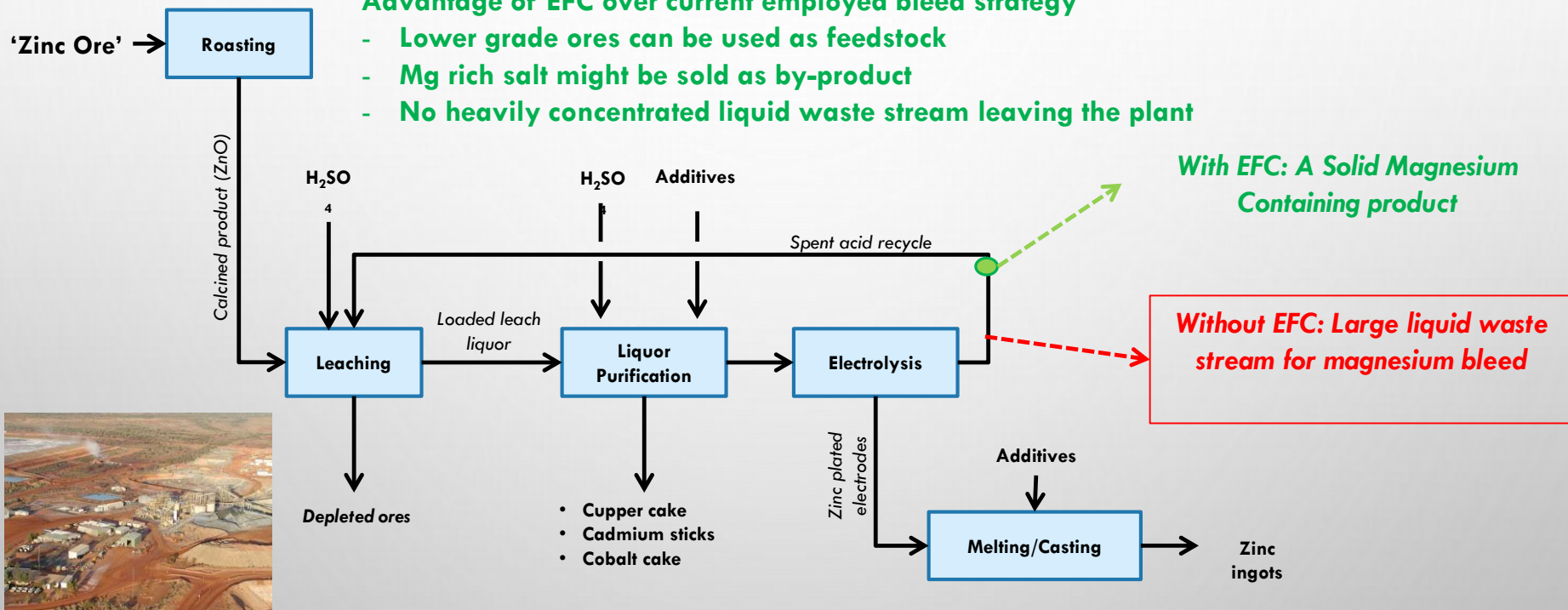


METAL ORE LEACHATE TREATMENT

Project for a large metal producer in The Netherlands

Advantage of EFC over current employed bleed strategy

- Lower grade ores can be used as feedstock
- Mg rich salt might be sold as by-product
- No heavily concentrated liquid waste stream leaving the plant



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RECOVERY OF A FERMENTATION PRODUCT

Project for a major engineering firm/process developer in Germany

Fermentation process



- No risk for thermal degradation of the product.
- Less biological activity in downstream processing system due to low temperature.
- High purity crystal product inherent to crystallization behaviour at low temperature operation.
- Flexible, easy capacity increase by modular EFC process design.

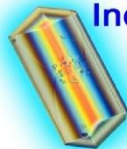
Filtration

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Clean water product (internal recycle)

Concentrated liquid stream (further treatment, C-rich by-product)

Crystalline organic acid product



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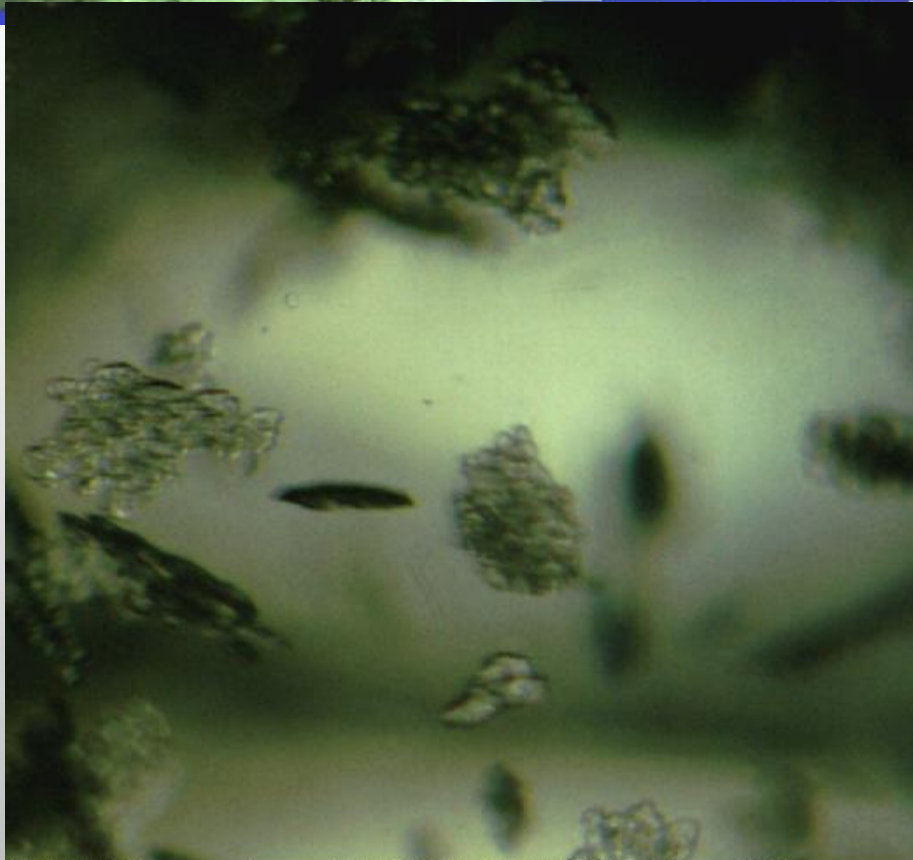
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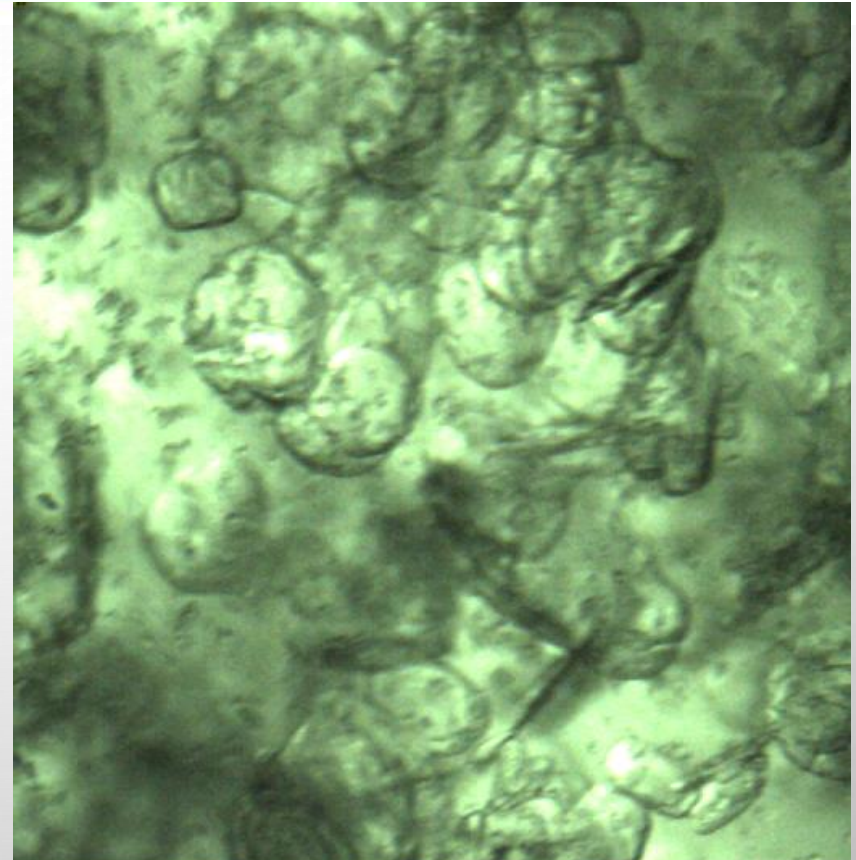
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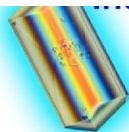
Purity of Ice and Salt Products



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#00182, V3.6.2



5/29/2014 12:30:13 PM 3414 -000.210ms 520x540, 1929 Hz, 513 μ s, *1,
Mikrotron #00182, V3.6.2



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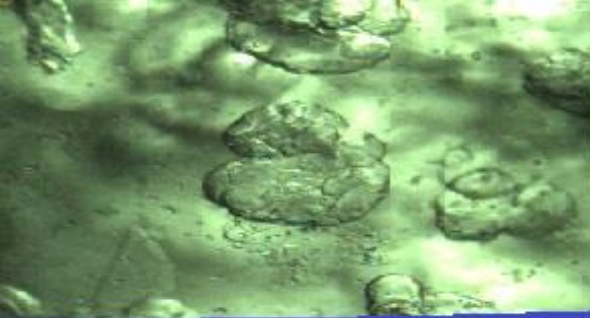
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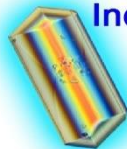
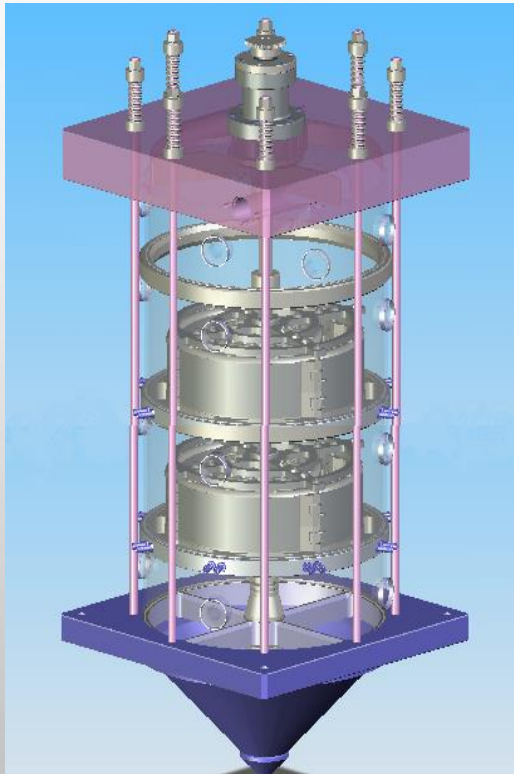
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SEVERAL PATENTED CRYSTALLIZERS



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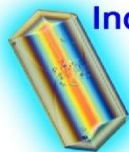
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Salt and Ice cake on the belt filters



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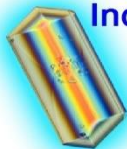
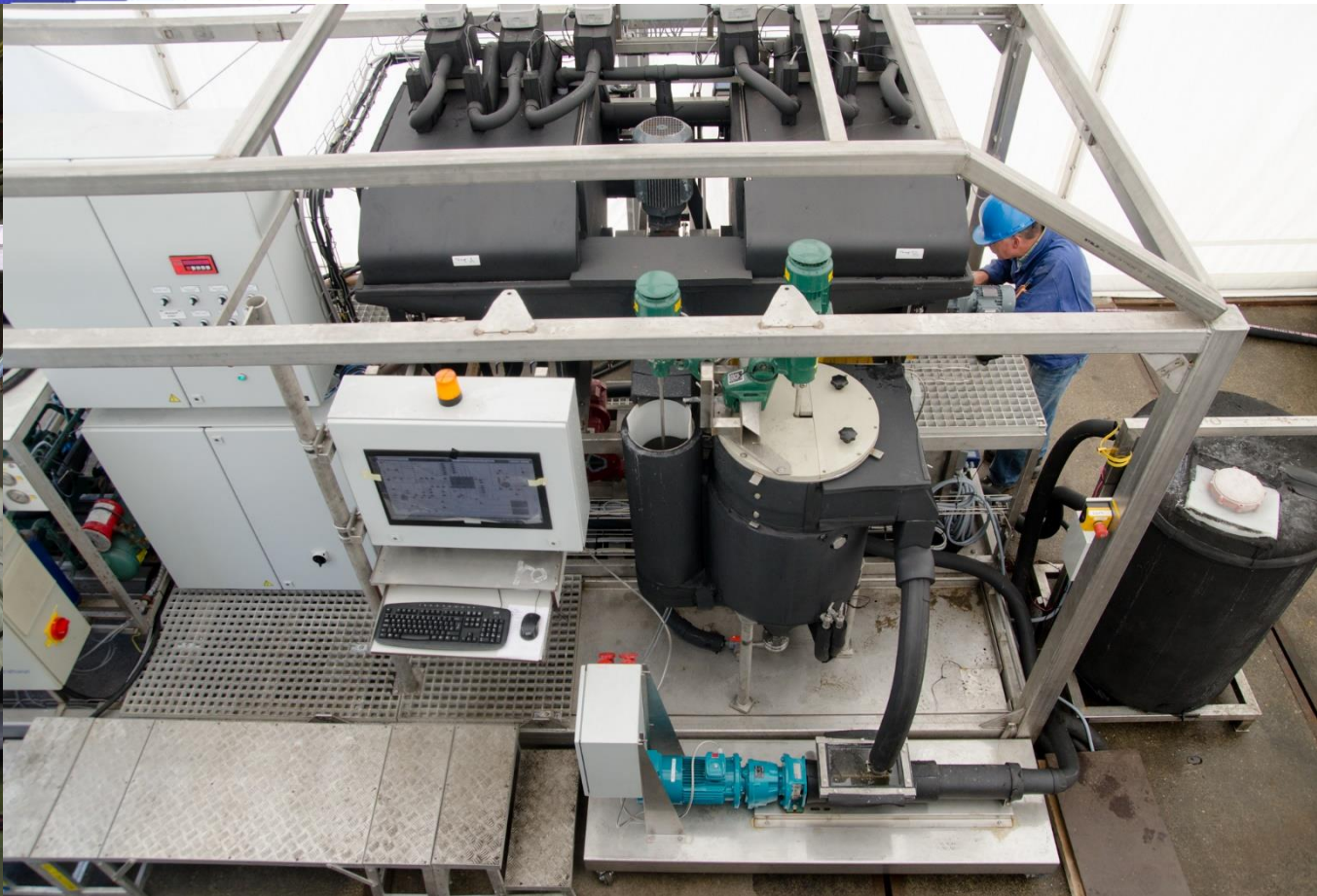
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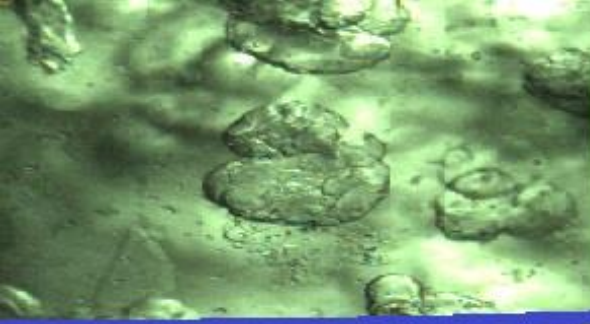
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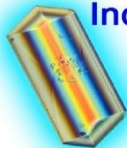
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Tested and demonstrated in Industrial environment



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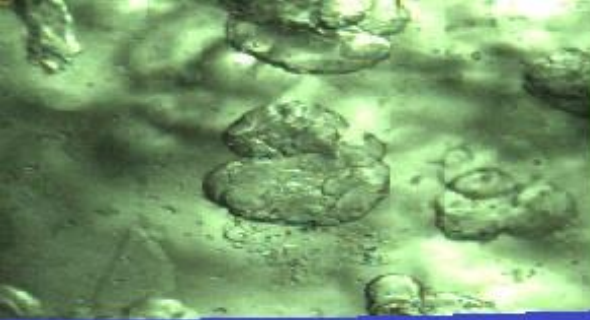
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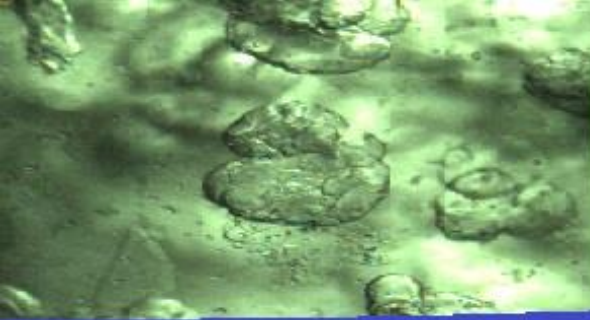
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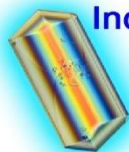


Conclusions

- Eutectic Freeze Crystallizers have been, and continue being, implemented to perform in an industrial scale level.
- Eutectic Freeze Crystallization is a sustainable environmentally friendly technology capable to work towards Zero Waste, not only cleaning water but producing valuable sellable products.
- Economic comparisons with existing brine water treatment technologies indicate that Eutectic Freeze crystallization alone or integrated is viable and produce a higher and alternative revenue.



Thanks for your attention



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