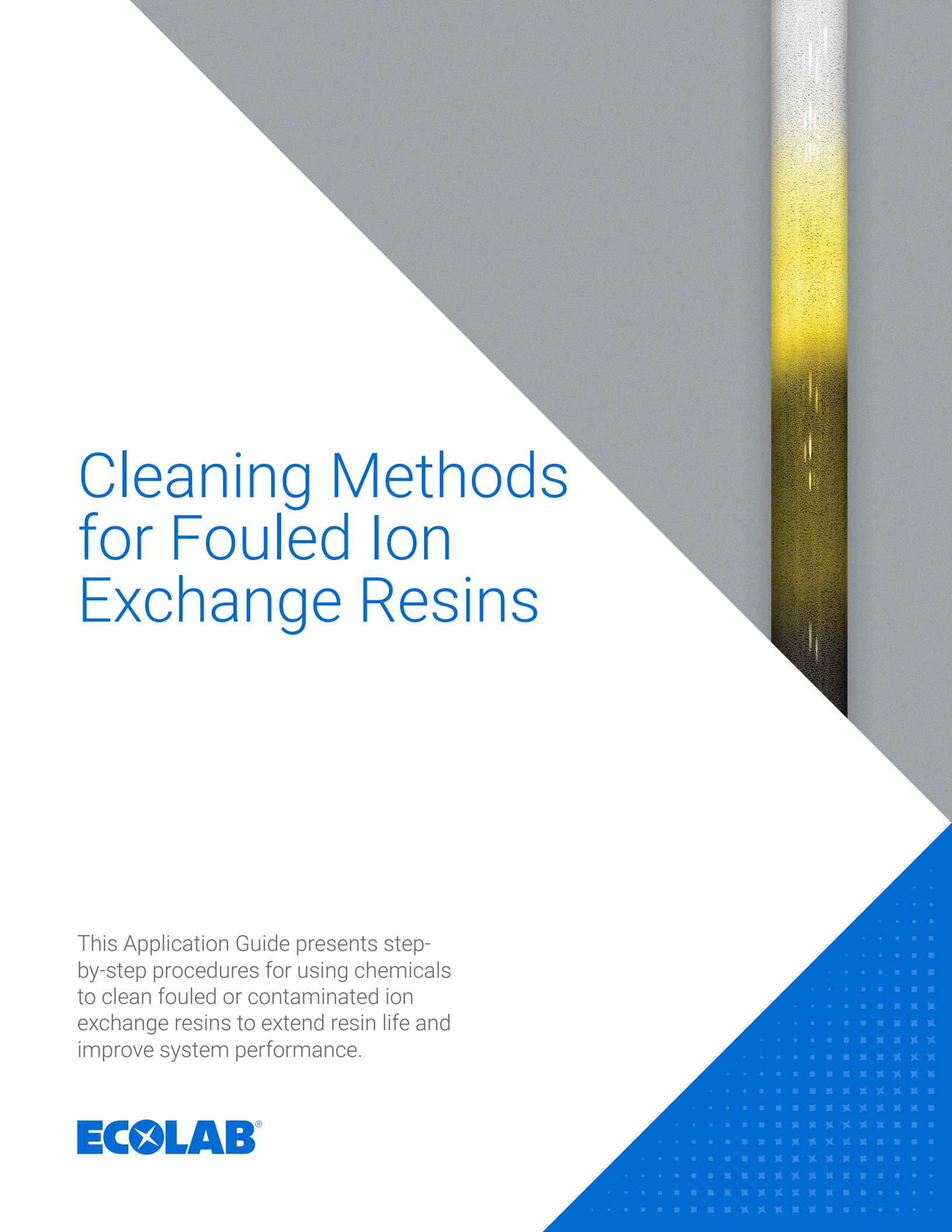




Cleaning Methods for Fouled Ion Exchange Resins

This Application Guide presents step-by-step procedures for using chemicals to clean fouled or contaminated ion exchange resins to extend resin life and improve system performance.



Cleaning Methods for Fouled Ion Exchange Resins

This Application Guide presents procedures for cleaning fouled or contaminated ion exchange resins for improved system performance. For more detailed information, visit www.purolite.com or contact the closest Purolite regional office listed on the back cover.

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NOTE: Metric measurements are accurate; US measurements are estimated conversions.



Most types of ion exchange resins can become chemically fouled or contaminated with suspended solids.

Precipitations can occur as a result of changes in pH, and/or concentration of relatively insoluble salts. Adsorption or ion exchange of other chemical elements that are not easily removed by the normal regeneration procedures can cause gradual fouling of the resin. Regular cleaning treatment can reduce fouling and extend resin life.

Increased regenerant quantities, regeneration frequency and elevated regeneration temperature may reduce fouling by preventing foulants from gaining a permanent hold.

A good operating practice is to ensure that all resin in the ion exchange units is regularly contacted with sufficient regenerant and subject to a regular cleaning procedure.

Causes of Deteriorating Performance in an Ion Exchange Plant

Despite precautions taken, the following conditions can cause deterioration in plant performance.

- Loss of resin from the operating units.
- Change in feed analysis. If the concentration of influent water chemistry is increased, throughput will be reduced proportionately. It is good practice to check the feed analysis regularly and make adjustment to accommodate any change.
- Faults in the operating process. Incorrect conditions of regeneration, failure to operate the plant according to instructions, faulty backwash procedures, and inconsistent rinse volumes and regenerating concentrations are relatively easy to ensure all functions are operating correctly.
- Malfunction of the plant engineering hardware. This is the most difficult condition to diagnose — requiring unit shut down, removal of the resin and thorough inspection and testing.
- Resin fouling or degradation.

If bed levels, water chemistry, operating processes and engineering hardware check out satisfactorily, a sample of resin should then be examined.

A bed volume (BV) is the amount of resin that needs to be treated. For example, if the vessel holds 100 ft³ of resin, that is the BV.

Evaluating Resin Through Sampling

Results of resin analysis will depend upon how the sample is taken. It is preferable to ensure that the sample collected is representative of the bulk quantity of the bed. If this is not possible, the sampling method should be supplied to the analyst.

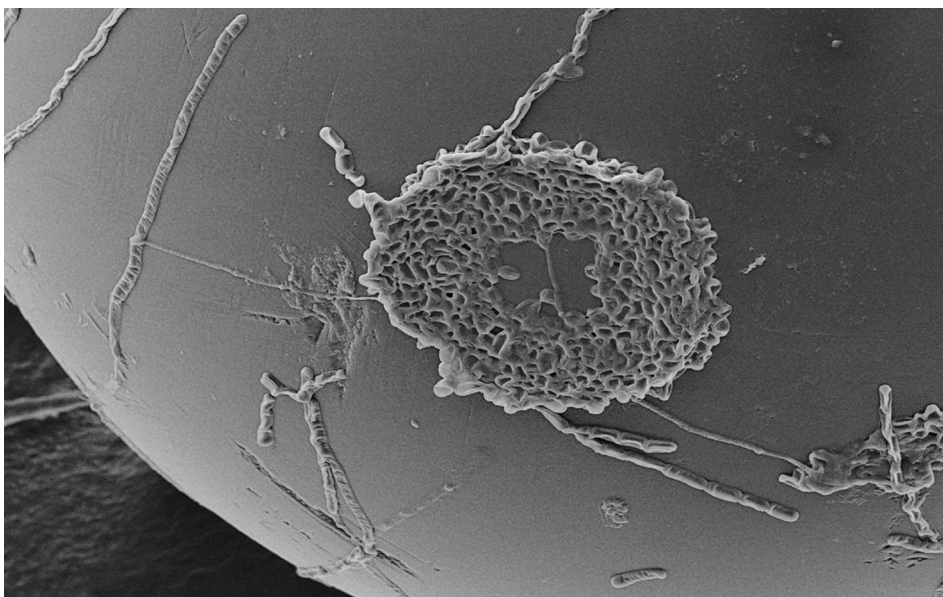
To obtain a representative sample of the resin contents of a mixed bed, please refer to our brochure on this subject at <https://bit.ly/3zg84kX>.

Analyses will report aging and fouling of sampled resin. If required, various procedures are available to clean resins. This document covers cleaning procedures for the following:

- Fouling by bacteria and algae
- Fouling by iron and manganese
- Fouling by organic species
- Fouling by oil

Disinfection Treatment Procedures for Bacteria and Algae

Under certain conditions, resin can become fouled either by bacteria or algae when contaminated water sources are fed to ion exchange systems. When contamination of resin beds is observed, consider the following treatments.

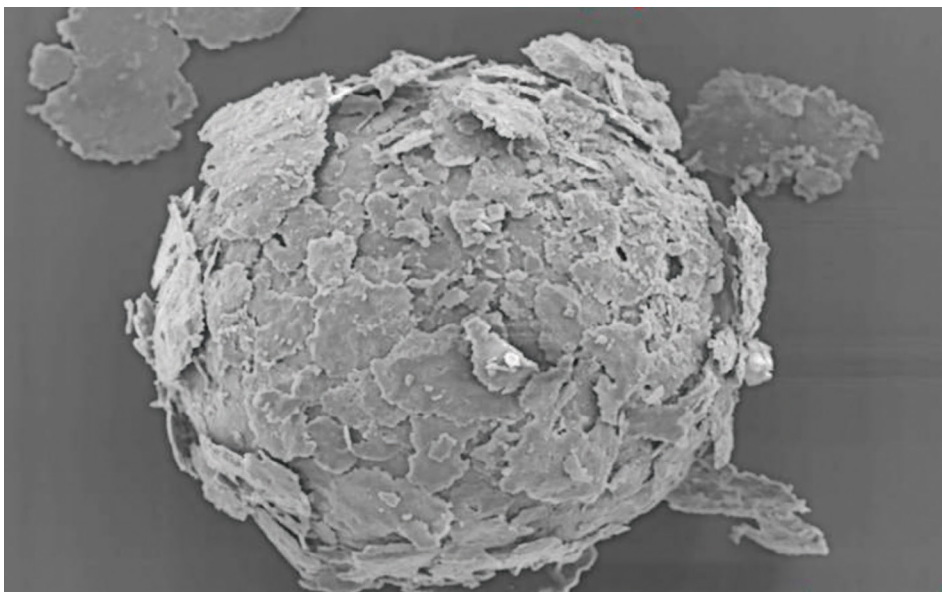


Bacteria contamination

Peracetic Acid for Non-Potable Systems

Peracetic acid, a derivative of hydrogen peroxide, is a good treatment against a wide variety of microbes. Research has shown that peracetic acid will be used to an ever-increasing degree in the field of medicine due to its anti-bacterial, fungicidal, sporicidal and anti-viral action.

Work done by the Degussa Technical Applications Department in conjunction with Chemiewerk Homburg AG determined that peracetic acid is suitable as a disinfectant for deionizers because of its wide spectrum of attack. Using a 0.1% peracetic acid solution in water with a reaction time of one hour, a slime and mold concentration of 10^4 – 10^5 /mL was reduced to almost zero. The short rinsing time after using peracetic acid is of importance (typically about 45 minutes or 10–15 BV).



Scaling fouled resin

Experiments have shown that in addition to excellent disinfection action, peracetic acid has minimal effect on the ion-exchange properties of cation or anion resins.

If peracetic acid is used as a disinfectant, the following procedure should be used for both cation and anion resin in regenerable systems.

- Ensure anion resins are fully exhausted as peracetic acid performs best at a pH < 8.
- Make up 1 bed volume (BV) of peracetic acid solution containing 0.1% peracetic acid.
- Inject 1 BV of disinfectant at a flow rate of 5 BV/h (0.6 USGM/ft³) with displacement discharged to a drain approved for chemical waste disposal.
- When all the peracetic acid has been injected, close all valves and retain the disinfectant for at least one hour to soak the resin and pipe work.
- Carry out a displacement rinse using raw water for at least 60 minutes at 5 BV/h, followed by a fast flush for 30 minutes.
- Regenerate the resin once and return the unit to service.

Reducing Bacteria in Single-Use Potable Anion Ion Exchange Resin Treatment Systems

The first step is always to try to locate the source of the bacteria. Perform bacterial testing at the well head, after the filter housings, and at any other inputs the system. It is impossible to eliminate bacteria from the resin if the influent to the resin bed has bacteria.

Once the influent source is eliminated, the next step is to flush the system out. Often, a 24-hour flush at design flow is enough to clean up a system adequately to pass bacteria tests. If a 24-hour flush at full flow is not feasible due to site conditions, try to flush as much volume as reasonably possible, and pull bacterial samples.

Please note — single-use resin systems should not be backwashed. If there is enough biological activity to cause clogging, evaluate if solids can be physically removed from the top of the vessel with a vacuum or a net. Make sure all equipment is sterilized before use to ensure no more bacteria is introduced into the system.

If a flush is not successful, chemical treatment may be necessary.

Chlorine should never be used with anion resin as a disinfectant for several reasons. First, the chlorine is consumed before a chlorine residual can be established throughout the bed, so the full bed is not disinfected. While the chlorine is consumed, the chlorine is also breaking chemical bonds in the resin. Functionality of the resin is lost, and chemicals like nitrosamines will be formed and released.

For these reasons, hydrogen peroxide is more commonly used as it is less oxidative than chlorine, yet it still has oxidative and biocidal efficiency necessary to get a system back in service. Use only Food Grade ANSI/NSF 60 certified products for potable water systems.

To disinfect a drinking water system using a single-use anion ion exchange resin, follow these steps.

- Before hooking up any equipment or hosing, ensure it has been disinfected and rinsed.
- Drain about one third of the water that is above the resin in the vessel to leave freeboard space in the vessel for any foaming that may occur.
- Establish a vent path (for possible foaming) with a resin screen to prevent resin spillage
- Determine the amount of peroxide need to create a solution of 0.1% or 1000 ppm of hydrogen peroxide.
- The basis of the volume of solution will be around half the total volume of the vessel.
- Contact Purolite if help is required.
- Start recirculation of the vessel with a flow rate of 5 BV/h (0.6 USGPM/cu ft) discharged to a drain approved for chemical waste disposal.
- Inject the hydrogen peroxide into the system, preferably in the recirculation pump suction.
- Verify concentration of hydrogen peroxide with test strips at the outlet sample port. Approach the injection slowly. Be careful not to overdose the peroxide into the resin bed.
- Recirculate the system for a minimum of one to two hours. Check the concentration periodically.
- Rinse or flush the vessel to a zero residual of hydrogen peroxide at the outlet sample port, discharging to a drain approved for chemical waste disposal.
- This is typically about 45 minutes or 10–15 BV when flushing at full flow.

- Pull bacteria samples
- Continue to flush, if possible, while awaiting sample results. If continuous flushing is not achievable, the resin should be flushed a minimum of 5 BV's at a flowrate greater than 6 USGPM/SQFT every 24 hours.
- CAUTION: HYDROGEN PEROXIDE HAS A HIGH OXIDIZING POTENTIAL. EXCESSIVE DOSING COULD RESULT IN IREVERSABLE RESIN DAMAGE. THE USER ASSUMES ALL RESPONSIBILITIES FOR THE EXECUTION OF THIS GUIDE AND THE CHEMICAL APPLICATION.

Sodium Hypochlorite

Sodium hypochlorite is widely available in the form of small carboys and containers. For resin sterilization, a 0.1% available chlorine solution should be used. This is obtained by diluting the commercially available hypochlorite.

To prepare the treatment, adhere to the following procedures.

- Regenerate the column with brine before treatment to convert all resin to the exhausted form (a double or triple regeneration is often required.) Note that cation resin will produce chlorine gas if not properly exhausted before treatment.
- The minimum volume of solution required to treat the bed is 3 BV (i.e. 3 times the resin volume installed in the unit).
- Pass the first bed volume through the bed at normal regeneration flow rate, or approximately 4 BV/h (0.5 USGM/ft³).
- Retain a portion of the second bed volume in the bed for no more than 2 hours.
- Pass the third bed volume through the bed at approximately 4 BV/h.
- Displace the sodium hypochlorite at a rate of approximately 4 BV/h with softened water, then rinse thoroughly to remove any trace of sodium hypochlorite. At least 8–10 bed volumes are required.
- Triple regenerate the resin before returning to service.

Note that this form of treatment may slightly break down the cross-linked matrix of the resin. As such, frequent treatments are not advised.

The procedure is also not recommended for phenolic, polycondensation and chelate resins.

For anion resins, the oxidizing effect of the sodium hypochlorite is on the amine groups, and therefore disinfection in sodium hypochlorite should only be considered in extreme cases on a once-off basis.

Suitable safety precautions should always be taken when using sodium hypochlorite. Additionally, all environmental laws and regulations should be followed when discharging waste into drains. All discharge areas should be free from acids or other chemicals that may react adversely with the dilute hypochlorite discharge.

Treatments for Iron and Manganese Fouling

Iron is present in several different forms within water. For example, in the case of un-aerated borehole water, iron can be present in the ferrous form (Fe^{++}), but on oxidation, it is converted into the ferric form (Fe^{+++}).

Iron can also be complexed with organic matter; in which case it is present as an anionic complex.

Normally, iron present in the ferric state is removed by cation resin operated in either the sodium or hydrogen forms.

In the case of hydrogen form cation resin representing the first stage of a demineralization system, the iron is removed from the water and eluted on regeneration with mineral acid. The situation is different with softening resin. Here, the ion exchange resin removes the iron from the water, but the regeneration procedure — which uses brine — does not elute the accumulated iron from the resin during the regeneration cycle. Consequently, the iron accumulates on the resin from cycle to cycle and steadily causes progressive iron fouling.

In the case of iron being present as organic/iron complexes, the complex is present as an anion, and is removed from the solution by the anion resin.

Because anion resin is regenerated with caustic soda, the iron is retained on the resin even though organic material is substantially removed with each regeneration cycle. This accumulation of iron on the resin causes the anion resin to become iron fouled.

When iron content of water is $> 0.5 \text{ mg/L}$ (0.5 ppm), it is recommended that some form of pre-treatment is used to reduce the iron level to $< 0.1 \text{ ppm}$.

The use of sulfuric acid while iron is present in feed water can result in an accumulation of iron on the resin and cause reduction in performance. In these cases, treatment with hydrochloric acid should be considered providing the internal construction of the units and attendant pipe work are compatible with hydrochloric acid.

In cases where iron accumulates on softening resin, either hydrochloric acid or citric acid treatment can be considered for treatment. When iron content of water is > 0.5 mg/L (0.5 ppm), it is recommended that some form of pre-treatment is used to reduce the iron level to < 0.1 mg/L (0.1 ppm). An alternative option is to use special Purolite resins, called Shallow Shell technology — or SST®. The SAC or WAC versions are able to treat water with up to 20 mg/L (20 ppm) of Fe^{2+} .

Removal of Iron from Cation Exchange Resin Using Citric Acid

On-Line Cleaning

Iron and calcium fouling of both weak and strong acid cation resins (WAC and SAC) can be prevented by treating the resin periodically with citric acid. Commercially available fifty percent (50%) citric acid is available in food grade form and can be used for resin that is used for softening in potable water as well as for food and beverage related applications. When working with any acidic material follow all precautions listed on the product's MSDS.

Online cleaning with citric acid is a simple procedure and requires a chemical feed system comprised of a small tank and a chemical feed pump. 50% citric acid is used for both on-line and off-line cleaning procedures.

In the case of a WAC or SAC resin operating in the hydrogen form (acid regeneration), the solution is pumped into the backwash inlet at a dosage of 250 ml/cubic foot of resin($\text{ft}^3\text{-R}$). In the case of a SAC operating in the sodium form (salt regeneration), the same dosage is used, but the citric acid is metered into the brine dilution water or add the full amount of citric acid into the brine tank.

The pump rate is established based on the dosage and the time to complete the backwash step (or dilute brine feed if using a SAC in the sodium form). For example, 100 ft^3 of resin will require 25 liters of 50% citric acid. If the backwash step takes 15 minutes, the feed rate should be set to inject the citric acid over the entire course of the backwash step. In this example, the pump would be set at 1.67 liters/min. A minimum contact time of 30 minutes is typical for SAC softeners, so in this case the feed rate would be set at 0.83 liters/min.

The frequency of treatment is based on the influent iron concentration.

Raw Water Iron Treatment Frequency

< 1 ppm every 6th regeneration

1–2 ppm every 4th regeneration

> 3 ppm every other regeneration

Off-Line Cleaning

Off-line cleaning with citric acid is prescribed when the resin is extremely fouled. This procedure requires the unit to be out of service for approximately eight hours. A mixing vessel, drum pump, air lance, and proper safety equipment will be required to perform this cleaning procedure.

Prior to cleaning the resin should be backwashed and regenerated as per normal operating procedures. This step will help reduce the amount of insoluble and soluble iron in or deposited on the resin and the tank internals.

The dosage for an off-line cleaning is 1.9 liters/ ft³-R of resin. In the previous example we used 100 ft³ of resin, so the off-line procedure would require 190 liters of 50% citric acid solution.

Open the vessel man way and drain the water to 12" above the top of the resin bed. Utilizing a drum pump, slowly pump the entire 190 liters of cleaning solution into the vessel. Drain the vessel again until the liquid level is approximately two inches above the top of the resin bed. This step places the cleaning solution in direct contact with the resin.

With the citric acid solution in contact with the resin bed, air-lance the entire resin bed using clean oil free plant air. A one-inch diameter PVC pipe can be used for the air lance. If the vessel has a gravel underdrain support, make sure to avoid contact with the air lance. The air-lancing step should be conducted for 2 -3 minutes every ½ hour for 4-6 hours.

Upon completion of the cleaning the vessel should be closed, filled, vented and backwashed with finished water until clear. After backwashing perform a standard regeneration.

In some circumstances, it may be difficult to remove the control head from the softener to add the citric acid and sparge the resin/cleaning solution. In this case, the saturated brine tank can be used to prepare the citric acid. Please note that it would be acceptable to mix the 50% citric with the saturated brine.

Use the system controls to add the cleaning solution to the resin. Be sure to empty the contents of the saturated brine tank to assure that all of the cleaning solution goes into resin. Soak as described above for 4 to 6 hours followed by a standard NaCl regeneration.

Citric Acid Cleaning Procedure for Purolite Strong Base Anion (SBA) Operated in Chloride (Cl⁻) Form Resin — Dealkalizing, or Organic Trap

Use of citric acid for restoring capacity of fouled ion exchange resin by removal of organic foulants, precipitated calcium/magnesium as well as iron and copper oxides.

1. Perform backwash of resin bed.
2. Regenerate with 160 g/L-R (10 lb/ft³-R) of 10% brine solution at 2 LPM/L-R (0.5 USGPM/ft³-R).
3. Perform thorough rinse of resin bed.
4. Drain the water level to 6" above resin with regenerated resin bed.
5. For 40% citric acid, add 70 ml/L-R (0.5 USG/ft³-R). For example: SBA tank contains 1,000 liters (35 ft³) = add 70 liters (20 USG) to resin bed.

For 90% citric acid, add 25 ml/L-R (0.2 USG/ft³-R). For example: SBA tank contains 1,000 liters (35 ft³) = add 25 liters (7 USG) to resin bed.
6. Drop water level to 2.5 cm (1 in) above resin bed (reducing resin level 13 cm (5 in) will allow reaction in under bedding).
7. Using air sparging technique, add induced air for 2–3 min. per ½ hour for a minimum of 8 hours. Disruption towards the bottom of the bed in an upward directional air flow will create foam and carbon dioxide (CO₂ from citric acid + calcium carbonate reaction). This would indicate suspected fouling at bottom of bed. You may be able to run the air for longer periods as the hardness breaks and foaming occurs in less volume.
8. Rinse for at least 30 min. or till effluent water is clear from vessel.
9. Regenerate the unit using a 10% NaCl and 2% Caustic Soda solution of 192–240 g/L (12–15 lb/ft³) at 2 LPM/L-R (0.5 USGPM/ft³-R) and let resin soak for 8 hours.
10. At end of soak, add 75–85 mL/L-R (7-8 USG/ft³-R) of same solution.
11. Perform typical rinse cycle of resin bed.
12. Double caustic regeneration dosage to convert from citrate form.

Preventative treatment going forward: Add 3–4 liters (3–4 quarts) 90% citric acid per 100 liters (3.5 ft³) of resin and leave out caustic during this regeneration.

Citric Acid Cleaning Procedure for Iron (Fe^{+2}) Removal From Purolite Strong Base Anion (SBA) Operated in Hydroxide (OH^-) Form Resin — Demineralizing

Use of citric acid for restoring capacity of fouled ion exchange resin by removal of iron and copper oxides.

1. This procedure is best done on an exhausted SBA resin bed.
2. Perform backwash of resin bed, but do not regenerate.
3. After regeneration is done, open the top manway and drain the water level down to 15 cm (6 in) above the resin bed.
4. For 40% citric acid, add 70 ml/L-R (0.5 USG/ft³-R). For example: SBA tank contains 1,000 liters
(35 ft³) = add 70 liters (20 USG) to resin bed.

For 90% citric acid, add 25 ml/L-R (0.2 USG/ft³-R). For example: SBA tank contains 1,000 liters
(35 ft³) = add 25 liters (7 USG) to resin bed.
5. Using Air Sparging technique, add induced air for 2–3 min. per ½ hour for a minimum of 8 hours. Disruption towards the bottom of the bed in an upward directional air flow will create foam and carbon dioxide (CO_2 from citric acid + calcium carbonate reaction). This would indicate suspected fouling at bottom of bed. You may be able to run the air for longer periods as the hardness breaks and foaming occurs in less volume.
6. Rinse for at least 30 min. or till effluent water is clear from vessel.
7. Repeat step 4 and let resin soak for 8 hours.
8. Perform fast rinse cycle of resin bed.
9. Perform a caustic regeneration cycle, hold the caustic inject step for 2x the normal time setting.

Treatments for Organic Fouling

Anion resins are susceptible to fouling by humic and fulvic acids sometimes found in surface waters.

These organic species become trapped within the resin matrix due to their large molecular weights. Symptoms of organic fouling include long rinse requirements, poor capacity and in the case of strong base resins, higher silica leakage.

The following treatment helps return the original ion exchange properties of the resin.

- Treat the resin at the end of the normal exhaustion cycle.
- Prepare three bed volumes of 10% w/v brine solution containing 2% w/v caustic soda. Temperature of the solution should be between 95 °F (35 °C) and 140 °F (60 °C) to ensure optimal organic elution effect.
- Introduce one bed volume into the ion exchange unit at a flow rate not exceeding 2 BV/h followed by a second bed volume.
- Retain the second volume in the unit for as long as possible (minimum 4 hours). Periodically agitate the bed throughout the retention period.
- At the end of the retention period, the last bed volume of brine should be passed through the resin at a rate of 1 BV/h and the resin rinsed thoroughly with clean water until free from brine.
- Subject the resin to at least two complete regeneration cycles before returning online.

Hydrochloric Acid

Occasionally, the presence of iron is detected on the anion resin. This can arise from iron/organic complexes being present in the raw feed water. As an alternate to HCl, see Sodium Chloride (salt) section below.

In these cases, it is advisable to treat the anion resin with 6% hydrochloric acid immediately after the brine treatment. The procedure that should be followed is similar to that given for brining.

It is very important that all traces of hydrochloric acid are removed from the unit before introducing caustic soda regenerant, and that all parts of the unit that come into contact with the hydrochloric acid are compatible with and resistant to the acid.

Sodium Chloride

- A. Add one bed volume solution of 15% NaCl to the vessel and allow it to soak for 240 minutes. If the temperature of the solution can be raised up to 80 ° C (176 ° F), the soak time can be reduced to 120 minutes.
- B. Fast rinse the bed for 1.5 bed volumes. If hardness is still leaking above set point, repeat step A above.
- C. If hardness leakage after step B is acceptable, proceed with a 90-minute acid regeneration cycle to fully covert the resin to H⁺ form.



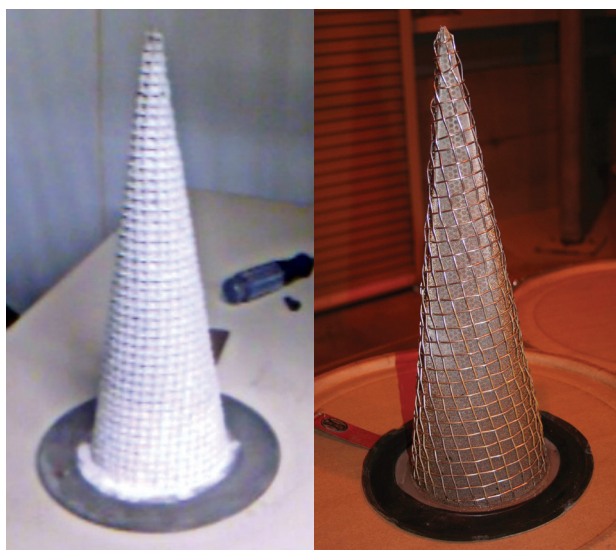
Iron fouling

Treatments for Oil Fouling

Oil in feed water or regeneration solutions will lead to fouling of ion exchange resins. Oil coats the surface of resin, making it difficult for ions to penetrate through the oil layer into the beads, where the majority of exchange sites are. Oil-based resin fouling results in deterioration of resin kinetics and treated water quality, as well as reduced operating capacity.

For best performance, there should be zero oil in feed solutions to the ion exchange resin bed.

Cleaning resins fouled by oil is extremely difficult. If resin is heavily fouled, it may be impossible to clean them sufficiently to make them suitable for ongoing use. The following procedure uses a low foaming, non-ionic



(Left) Pipeline strainer outlet of WAC 1 pre-cleaning with NaCl

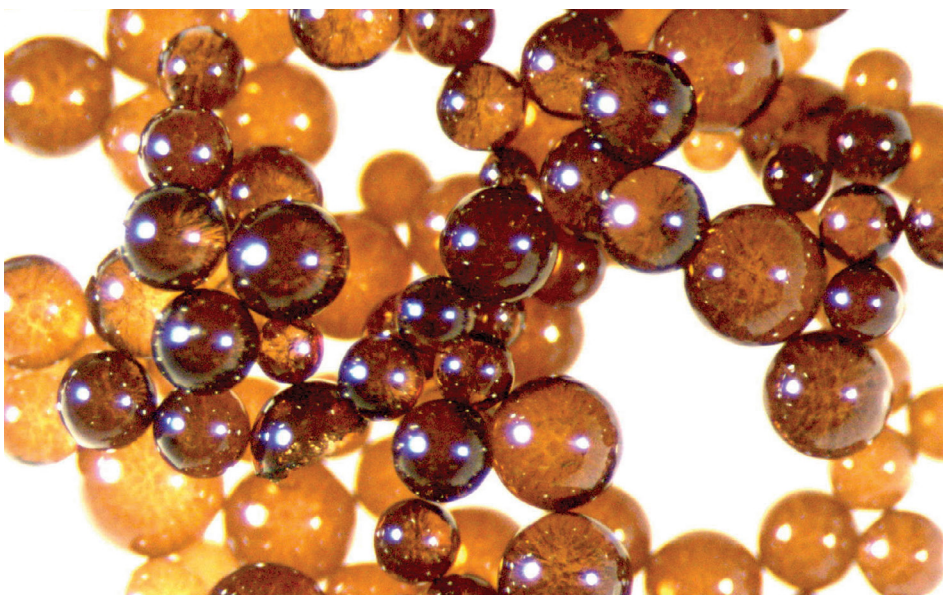
(Right) Clean pipeline strainer after cleaning with NaCl

surfactant and is recommended for lightly fouled ion exchange resins and inert polymers.

- Thoroughly backwash the fouled resin.
- Drain the unit and fill with a solution containing no more than 0.1% of surfactant. The treatment is most effective if the solution is administered at approximately 104 °F (40 °C). Using lower temperatures may produce considerable foaming. Note that it is important to use surfactant that does not foul resin.
- Cleaning will be more efficient if air is introduced to the resin bed as this causes agitation while the resin soaks in the surfactant solution. Agitation should continue for approximately a half an hour.

- Following this, thoroughly backwash the unit and rinse in down-flow mode until the foaming completely dissipates. Water temperature for the first part of the rinse is optimal at 104 °F (40 °C).
- Finally, thoroughly regenerate and rinse the resin before proceeding to the next service cycle.

Inspecting, sampling and cleaning the resin regularly can prevent the accumulation of contaminants and severity of resin fouling. This, in turn, will help optimize performance and prevent permanent degradation of the resin.



Oil fouled resin

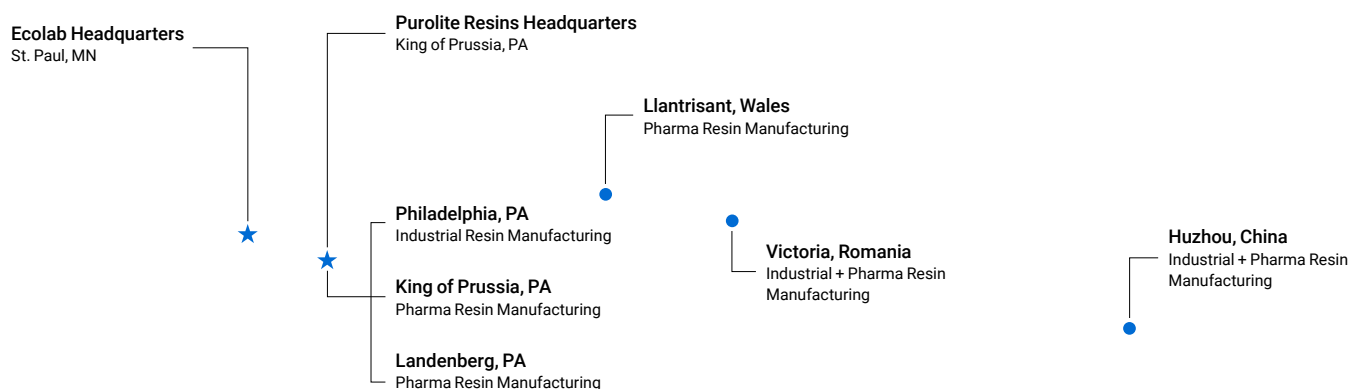
Abbreviations

BV	Bed volume
BV/h	Bed volume per hour
C	Celsius
cm	Centimeter
F	Fahrenheit
ft³	Cubic foot
ft³-R	Cubic foot of resin
g/L	Grams per liter
g/L-R	Grams per liter of resin
in	Inch
L	Liter
lb/ft³	Pound per cubic foot
lb/ft³-R	Pound per cubic foot of resin
LPM	Liters per minute
LPM/L-R	Liters per minute per liter of resin
L-R	Liter of resin
mg/L	Milligrams per liter
min	Minute
mL	Milliliter
mL/L-R	Milliliters per liter of resin
MSDS	Material safety data sheet
oz	Ounce
oz/ft³-R	Ounces per cubic feet of resin
ppm	Parts per million
SAC	Strong acid cation
SBA	Strong base anion
USG	United States gallon
USGPM	United States gallons per minute
USGPM/ft³-R	United States gallons per minute per cubic foot of resin
USG/ft³-R	United States gallons per cubic foot of resin
WAC	Weak acid cation
w/v	Weight per volume

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